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Quality Assurance Project Plan
for
Phase II Ground Water RCRA Facility Investigation Solid Waste Management Unit 30 (Landfarm)
Naval Surface Warfare Center
Crane, Indiana

Southern Division
Naval Facilities Engineering Command
Contract Number N62467-94-D-0888
Contract Task Order 0019

August 1999

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QUALITY ASSURANCE PROJECT PLAN FOR PHASE II GROUND WATER RCRA FACILITY INVESTIGATION SOLID WASTE MANAGEMENT UNIT 30 (LANDFARM)

NAVAL SURFACE WARFARE CENTER CRANE, INDIANA

COMPREHENSIVE LONG-TERM ENVIRONMENTAL ACTION NAVY (CLEAN) CONTRACT

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ACRONYMS/ABBREVIATIONS

B&R Environmental	Brown & Root Environmental
CLEAN	Comprehensive Long-Term Environmental Action, Navy
CLP	Contract Laboratory Program
CMS	Corrective Measures Study
COC	Chain-of-Custody
COEWES	Corps of Engineers Waterways Experiment Station
CVAA	Cold Vapor Atomic Absorption
DQO	Data Quality Objective
FOL	Field Operations Leader
ft	feet
FTMR	Field Task Modification Request
GC/MS	Gas Chromatography/Mass Spectrometry
HPLC	High-Performance Liquid Chromatography
HSWA	Hazardous and Solid Waste Amendments
ICP	Inductively Coupled Plasma
ICP/MS	Inductively Coupled Plasma/Mass Spectroscopy
IDEM	Indiana Department of Environmental Management
IDL	Instrument Detection Limit
IDW	Investigation Derived Waste
LCS	Laboratory Control Sample
LIMS	Laboratory Information Management System
µg/L	micrograms per liter
mg/L	milligrams per liter
MCL	Maximum Contaminant Level
MDL	Method Detection Limit
MS/MSD	Matrix Spike/Matrix Spike Duplicate
mS/cm	milli-siemens per centimeter
NSWC	Naval Surface Warfare Center
NEESA	Naval Energy and Environmental Support Activity
NFESC	Naval Facilities Engineering Service Center
NIST	National Institute of Science and Technology
NTU	Nephelometric Turbidity Units
ORP	Oxidation Reduction Potential

PARCC	Precision, Accuracy, Representativeness, Comparability, Completeness
PE	Performance Evaluation
PRG	Preliminary Remedial Goals
QA	Quality Assurance
QAM	Quality Assurance Manager
QAPP	Quality Assurance Project Plan
QC	Quality Control
RCRA	Resource Conservation and Recovery Act
RFI	RCRA Facilities Investigation
RL	Reporting Limit
RPD	Relative Percent Difference
SDG	Sampling Delivery Group
SOP	Standard Operating Procedure
SQL	Sample Quantitation Limit
SWMU	Solid Waste Management Unit
TKN	Total Kjeldahl Nitrogen
TOM	Task Order Manager
TtNUS	Tetra Tech NUS, Inc.
U.S. EPA	U.S. Environmental Protection Agency
VOC	Volatile Organic Compound
%R	Percent Recovery

1.0 PROJECT DESCRIPTION

This quality assurance project plan (QAPP) presents the organization, objectives, planned activities, and specific quality assurance and quality control (QA/QC) procedures associated with a Phase II Ground Water Resource Conservation and Recovery Act (RCRA) Facility Investigation (RFI) to be performed for Solid Waste Management Unit (SWMU) 30 located at the Naval Surface Warfare Center in Crane, Indiana (NSWC Crane). SWMU 30 is also referred to as the "Landfarm". Specific protocols for sampling, sample handling and storage, chain-of-custody, and laboratory and field analyses are described. All QA/QC procedures are structured in accordance with applicable technical standards, and U.S. Environmental Protection Agency (U.S. EPA) Region 5 requirements, regulations, guidance, and technical standards. The Naval Facilities Engineering Service Center (NFESC) guidance document entitled "Navy Installation Restoration Laboratory Quality Assurance Guide" (NFESC, February 1996) was also used in establishing the QA/QC requirements specified in this QAPP. This project description outlines the overall scope of the Phase II Ground Water RFI.

1.1 INTRODUCTION

This QAPP has been prepared by Tetra Tech NUS, Inc. (Note: Brown & Root Environmental [B&R Environmental] was purchased on January 1, 1998, and became Tetra Tech NUS, Inc. [TtNUS]) on behalf of the U.S. Navy Southern Division Naval Facilities Engineering Command and the NSWC Crane. This QAPP and other associated documents, including the Work Plan and Health and Safety Plan, constitute the project planning documents for the Phase II Ground Water RFI.

1.1.1 Overall Project Objectives

The objectives of this project are to determine the possible presence or absence and extent of ground water contamination at SWMU 30. This work is being conducted to meet the requirements of a Phase II Ground Water RFI.

Seven ground water monitoring wells were installed around the perimeter of SWMU 30 (the Landfarm) during a previous U.S. Army Corps of Engineers Waterways Experiment Station (COEWES) investigation. These wells have not been sampled to date. The scope of the current investigation involves sampling the seven on-site ground water monitoring wells five times over a 15-month duration, performing laboratory analysis of the samples, validating the analytical data, managing the data, and reporting the status and results.

Specific Data Quality Objectives (DQOs), as discussed in Section 1.4, were developed early in the document planning stage in order to accomplish the project objectives.

1.1.2 Project Status/Phase

The activities discussed in the three planning documents (QAPP, Work Plan, and Health and Safety Plan) constitute the first comprehensive environmental investigation of ground water at the Landfarm. Although a phased investigative approach is not planned at this time, the results of the environmental investigations outlined in the Work Plan will dictate the need and scope of follow-up environmental investigations.

1.1.3 QAPP Preparation Guidelines

This QAPP has been prepared in accordance with specific Region 5 guidance for this project and the general guidance outlined in the U.S. EPA "Region 5 QAPP Policy" (U.S. EPA Region 5, April 1998). Additional guidance regarding the QAPP contents relative to the current project was provided by the U.S. EPA Region 5 Remedial Project Manager during teleconferences held on June 3, 1998, and February 19, 1999. Representatives of U.S. EPA Region 5, NSWC Crane, the U.S. Navy Naval Facilities Engineering Command (Southern Division), and TtNUS participated in the teleconferences.

1.2 SITE BACKGROUND AND DESCRIPTION

NSWC Crane, including its location, size and borders, regional geology, hydrogeology, hydrology, and topography, is briefly discussed in the remainder of this section. Most of the information is contained in the Work Plan for the SWMU 30 Phase II Ground Water RFI, and specific sections of the Work Plan are referenced as appropriate.

1.2.1 Location

NSWC Crane is located in Crane, Indiana, approximately 75 miles southwest of Indianapolis, Indiana, and 71 miles northwest of Louisville, Kentucky. A site location map for the facility is provided as Figure 1-1 of the Work Plan.

1.2.2 Facility Size and Borders

NSWC Crane encompasses more than 100 square miles (64,463 acres). The facility is bordered on the east by private farmland, on the north by Highway 45, on the west by Highway 458 and U.S. 231, and on the south by private farmland.

1.2.3 Natural and Manmade Features

Detailed information regarding natural and manmade features at the site is provided in Section 1.2.3 of the Work Plan.

1.2.4 Topography

Detailed information regarding site topography is provided in Section 1.2.2 of the Work Plan.

1.2.5 Local Hydrology and Hydrogeology

Detailed information regarding regional geology, hydrogeology, and hydrology is provided in Sections 1.3.1 and 1.3.2 of the Work Plan.

1.3 FACILITY HISTORY

Within the facility, NSWC Crane operates a wastewater treatment plant that generates sludges. NSWC Crane has historically used land application for the disposal of these sludges. The sludges are from the processing of domestic and process wastewater. Process sources include metal finishing operations; surface coating operations; the loading, assembling, and packing of ordnance; water treatment plant backwash; boiler blowdown; and industrial laundry operations.

In October 1980, NSWC Crane filed a RCRA Section 3010 Notification and a Part A (interim status) permit application to operate as a treatment, storage, or disposal facility. The application for the Part A permit was approved, and the facility was allowed to operate as though it had a permit. In December 1983, NSWC Crane applied for and obtained a sludge application permit to spray sludges from its wastewater treatment plant along approximately 18 miles of roadside near the western boundary of the facility. In February 1988, NSWC Crane was issued a permit to apply sludge to a 2.5-acre site, the Landfarm (see Figure 1-2 of the Work Plan), located near an existing sanitary landfill. Sludges from the on-site wastewater treatment facility were applied to this site from 1988 through 1993. In June 1994,

NSWC Crane began applying sludges to eight new land-application-permitted sites located south and southeast of the Landfarm.

On December 20, 1989, a joint federal and state RCRA storage permit was issued to the Navy. The federal portion of the permit established the Hazardous and Solid Waste Amendments (HSWA) Corrective Actions Requirements and Compliance Schedules (RCRA 3004). The compliance schedule obligated NSWC Crane to perform an RFI at each on-site SWMU. A Corrective Measures Study (CMS) and implementation of corrective measures are to be initiated if contamination is identified at a SWMU during the RFI process. The State of Indiana obtained pre-HSWA authorization and issued the Navy the state portion of the permit.

In 1992, U.S. EPA became concerned that the sludges from the on-site treatment plant should be characterized as F006 waste (wastewater treatment sludges from electroplating operations). U.S. EPA contended that wastewaters discharging from the electroplating shop pretreatment plants (Building 38 and 3064) were mixing with other wastewaters in the sewer system prior to arrival at the main sewage treatment plant. In response to U.S. EPA's concerns, NSWC Crane began managing wastes from the plating shops to prevent this discharge and resulting mixing. In 1995, U.S. EPA renewed and modified the NSWC Crane permit to include the Landfarm as SWMU 30 to settle the enforcement issue concerning F006 waste. Included in the modification were corrective action requirements due to HSWA. U.S EPA has required NSWC Crane to conduct an RFI at the Landfarm to determine if the previous application of sludges, which were possibly contaminated with plating wastes, have adversely affected the shallow ground water regime.

1.3.1 General History

The general history of the NSWC Crane and the Landfarm is provided in Section 1.2.1 of the Work Plan.

1.3.2 Past Data Collection Activities

Section 1.3 of the Work Plan summarizes the environmental investigations conducted to date.

1.3.3 Current Status

The Landfarm at NSWC Crane is no longer used for sludge application. In 1995, U.S. EPA reviewed and modified the NSWC Crane permit with the stipulation that an RFI be conducted at the Landfarm to determine if the application of sludges possibly contaminated with plating wastes have affected the shallow ground water regime. If statistically significant increases in contamination are found using 40

CFR 264 subpart F evaluation procedures outlined in the Work Plan, NSWC Crane will be required to conduct a CMS.

1.4 PROJECT OBJECTIVES

This section outlines the overall approach to meet the project objectives for the SWMU 30 Phase II Ground Water RFI at NSWC Crane. Specific objectives and associated tasks are discussed in Section 1.4.1. Project target parameters and intended data uses are discussed in Section 1.4.2. DQOs are discussed in Section 1.4.3. The SWMU 30 RCRA ground water monitoring requirements are summarized in Table 1-1.

1.4.1 Specific Objectives and Associated Tasks

Specific objectives for this RFI are summarized in Table 1-1 of this QAPP. Decision rules are also provided in Section 1.4 of the Work Plan.

1.4.2 Project Target Parameters and Intended Data Uses

This section discusses the field and laboratory analytical information to be generated during the course of the RFI. Field parameters and intended data uses are discussed in Section 1.4.2.1. Laboratory parameters and intended data uses are discussed in Section 1.4.2.2.

1.4.2.1 Field Parameters

Field parameters will include those associated with ground water sampling and analysis; they will be measured using simple field instrumentation. Field measurements of airborne volatile organic compounds (VOCs) will be obtained using a photoionization detector. These measurements will be used primarily for health and safety monitoring.

Field testing for several parameters, including dissolved oxygen, pH, specific conductivity, oxidation reduction potential (ORP), turbidity, water level, and temperature, will be completed for all ground water samples. These measurements will be used to support monitoring well development and purging of stagnant water from well casings. Specific conductivity and pH will also be used as general indicators of water quality. Hexavalent chromium (Cr^{+6}) will also be analyzed in the field using a Hach test kit, method 8023 (see Appendix E of the Work Plan). AccuVac ampuls will be used with a detection limit of 10 micrograms per liter (µg/L) (Cr^{+6} criterion is 180 µg/L).

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TABLE 1-1

SUMMARY OF LANDFARM (SWMU 30) RCRA GROUND WATER MONITORING REQUIREMENTS
NAVAL SURFACE WARFARE CENTER
CRANE, INDIANA

Type of RCRA Program and Objectives	Analytical Program (All Wells)				Monitoring Points		
	Parameter Type	Sample Frequency and Target Constituents	Objective	Limit of Detection	Location	Objective	Evaluations to be Performed
Type: RFI Characterization & Corrective Action Requirements Objectives: • Monitor GW for absence or presence of hazardous constituents and determine concentrations if present (upgradient and downgradient wells). • Delineate site-specific extent of the GW contaminant distribution, if present (upgradient and downgradient wells).	Field	Quarterly • pH • Specific conductivity • Temperature • Turbidity • Dissolved oxygen • Hexavalent Chromium[4] • Oxidation Reduction Potential (ORP) • Water-level measurements	Determine if monitoring well water is equivalent to formation water. Evaluate general water quality. Determine GW flow direction	Refer to the Sampling and Analysis Plan for applicable limits.	• 2 Upgradient wells 030-GW-002 030-GW-003 • 5 Downgradient wells 030-GW-001 030-GW-004 030-GW-005 030-GW-006 030-GW-007	• Monitor quality of upgradient GW that has not been affected by operations of the unit. • Monitor quality of GW that may have been affected by operations of the unit.	Determine if statistically significant evidence of GW contamination exists by comparing upgradient concentrations to downgradient concentrations. Compare to risk-based target level (Table 1-2).
	Laboratory	Quarterly[1, 2] • VOCs • Explosives • Metals (total and dissolved) • Cyanide • Nitrate/Nitrite • Ammonia • Phosphorous • TKN	Monitor waste constituents and reaction products that indicate presence of hazardous constituents in GW attributable to operations.	Reporting limit low enough to meet Region 5 criteria for protection of human health[3].			Determine site-specific extent of GW contaminant distribution, if present.

GW Ground water
NA Not applicable
VOCs Volatile organic compounds
TKN Total Kjeldahl Nitrogen

1 The full suite of listed analytes will be analyzed for Rounds 1 and 2, followed by a more focused list for Rounds 3 through 5.
2 See Table 1-2 for specific chemicals and analytical methods.
3 Human health-based criteria consist of U.S. EPA Region IX Preliminary Remedial Goals (PRGs) for tap water and Safe Drinking Water Act Maximum Contaminant Levels (MCLs).
4 Field analyses using Hach test kit method 8023 (see Appendix E of Work Plan).

It is presently unknown if aquifer characterization has been completed for the Landfarm wells. Slug tests will be performed to determine hydraulic conductivities. If existing data on aquifer characteristics are not available, pumping tests will be conducted in conjunction with the first ground water sampling event.

1.4.2.2 Laboratory Parameters

Information regarding the types of chemicals present at the Landfarm based on generator knowledge was used to develop a focused analytical program. The suite of analyses for the first two sampling rounds of the NSWC Crane RFI includes Appendix IX volatiles (excluding 1,4-dioxane, acetonitrile, isobutyl alcohol, and propionitrile), explosives, Appendix IX metals (total and dissolved), ammonia, cyanide, nitrate/nitrite, phosphorus (total and dissolved), and total Kjeldahl nitrogen (TKN). The four aforementioned Appendix IX volatiles were excluded from the volatile target compound list because these compounds cannot be accurately analyzed using SW-846 method 8260B, and they are not expected to be present at the Landfarm based on generator knowledge. Analytical data from the initial two sampling rounds will be used to characterize the upgradient and downgradient ground water and will enable TtNUS to develop a list of potential contaminants of concern for this site. (Section 1.4 of the Work Plan provides further information regarding this process.) In turn, the parameter list for the three subsequent sampling events will be based on the list of potential contaminants of concern. Table 1-2 summarizes all target laboratory analytes and associated method detection limits (MDLs) or instrument detection limits (IDLs), as applicable, laboratory reporting limits (RLs), and risk-based target levels.

1.4.3 Data Quality Objectives

Ground water is being monitored for the following purposes:

- To comply with the U.S. EPA Region 5 Corrective Action requirement to perform an RFI at this SWMU.
- To determine whether any potential ground water contamination is related to operations previously conducted at this SWMU.

Proposed risk-based target levels based on previous U.S. EPA Region 5 guidance are presented in Table 1-2. The overall objective of SWMU 30 ground water monitoring is to meet requirements of the above-listed purposes, including the risk-based target levels for parameters listed in Table 1-2. This objective will be attained by performing the following activities:

TABLE 1-2

ANALYTICAL METHODS AND LIMITS OF DETECTION
NAVAL SURFACE WARFARE CENTER
CRANE, INDIANA
PAGE 1 OF 2

Chemical	Laboratory MDL/IDL[1] (ug/L)	Laboratory RL[1] (ug/L)	Risk-Based Target Level[2] (ug/L)	Source
EXPLOSIVES (SW-846 METHOD 8330)				
1,3,5-Trinitrobenzene	0.41	1.0	1100	prg
1,3-Dinitrobenzene	0.41	0.9	3.7	prg
2,4,6-Trinitrotoluene (TNT)	0.57	1.6	2.2 ca	prg
2,4-Dinitrotoluene	0.57	1.2	73	prg
2,6-Dinitrotoluene	0.83	2.0	37	prg
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	0.38	2.0	1800	prg
2-Nitrotoluene	0.48	2.0	370	prg
3-Nitrotoluene	0.48	2.0	370	prg
4-Nitrotoluene	0.48	2.0	370	prg
4-Amino-2,6-dinitrotoluene	0.35	1.6	--[3]	
2-Amino-4,6-dinitrotoluene	0.34	0.9	--	
Methyl-2,4,6-trinitrophenylnitramine (Tetryl)	0.29	1.2	370	prg
Nitrobenzene	0.22	1.5	3.4	prg
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	0.35	0.61[4]	0.61 ca	prg
APPENDIX IX METALS (SW-846 METHOD 6020 ICP/MS, UNLESS OTHERWISE NOTED)				
Antimony	1.0	1.0	6	mcl
Arsenic	1.0	1.0	2	[5]
Barium	0.5	1.0	2000	mcl
Beryllium	0.5	1.0	4 ca	[5]
Cadmium	0.5	1.0	5	mcl
Chromium (total)	0.5	5.0	100	mcl
Cobalt	0.5	3.0	2200	prg
Copper	0.5	2.0	1300	mcl
Lead	0.5	1.0	4	prg
Mercury (SW-846 Method 7470A)	0.06	0.2	200	mcl
Nickel	0.5	10	100[6]	mcl
Selenium	1.0	1.0	50	mcl
Silver	0.5	3.0	100 smcl	mcl
Thallium	0.5	1.0	2	mcl
Tin	0.1	10	22000	prg
Vanadium	0.5	2.0	260	prg
Zinc	5.0	10	11000	prg
MISCELLANEOUS PARAMETERS				
Ammonia (EPA Method 350.1)	6.4	10	[3]	
Cyanide (SW-846 Method 9012A)	2.4	10	200	mcl
Hexavalent Chromium (fixed Hach test kit method 8023)	[7]	[7]	180	prg
Nitrate/Nitrate (EPA Method 353.2)	2	10	10000	mcl
Phosphorus (Total and Dissolved) (EPA Method 365.2)	31.2	100	[3]	
Total Kjeldahl Nitrogen (EPA Method 351.2)	120	300	[3]	
APPENDIX IX VOLATILE ORGANIC COMPOUNDS[8] (SW-846 METHOD 8260B WITH 25 ML PURGE)				
1,1,1,2-Tetrachloroethane	0.13	0.5	5 ca	[5]
1,1,1-Trichloroethane	0.17	0.5	200	mcl
1,1,2,2-Tetrachloroethane	0.10	0.5	5 ca	[5]
1,1,2-Trichloroethane	0.14	0.5	5 ca	[5]
1,1-Dichloroethane	0.17	0.5	810	prg
1,1-Dichloroethene	0.12	0.5	5 ca	[5]
1,2,3-Trichloropropane	0.54	1[4]	0.0016 ca	prg
1,2-Dibromo-3-chloropropane	0.30	1	1 ca**	[5]
1,2-Dibromoethane	0.13	0.5	1 ca**	[5]
1,2-Dichloroethane	0.14	0.5	1 ca**	[5]
1,2-Dichloropropane	0.14	0.5*	0.16 ca**	[5]
2-Butanone	0.86	10	1900	prg
2-Hexanone	0.92	10	[3]	
4-Methyl-2-pentanone	0.52	10	160	prg
Acetone	1.8	10	610	prg
Acrolein	4.6	10*	4	[5]
Acrylonitrile	1.3	3	3.7 ca**	
Allyl chloride (3-chloro-1-propene)	0.22	10	1800	prg
Benzene	0.013	0.5	5 ca	[5]
Bromodichloromethane	0.13	0.5	100 ca	[5]
Bromoform	0.18	0.5	8.5 ca**	prg
Bromomethane	0.59	1[4]	8.7	prg

TABLE 1-2

**ANALYTICAL METHODS AND LIMITS OF DETECTION
NAVAL SURFACE WARFARE CENTER
CRANE, INDIANA
PAGE 2 OF 2**

Chemical	Laboratory MDL/IDL[1] (ug/L)	Laboratory RL[1] (ug/L)	Risk-Based Target Level[2] (ug/L)	Source
Carbon disulfide	0.13	0.5	1000	prg
Carbon tetrachloride	0.17	0.3[4]*	0.17 ca**	prg
Chlorobenzene	0.18	0.5	39	prg
Chloroethane (ethyl chloride)	0.21	0.5	8600	prg
Chloroform	0.14	0.3*	0.16 ca	prg
Chloromethane	0.17	0.5	1.5 ca	prg
cis-1,3-Dichloropropene	0.16	0.3[4]	[3]	
Dibromochloromethane	0.10	0.5	1 ca	prg
Dibromomethane	0.19	0.5	370	prg
Dichlorodifluoromethane	0.18	0.5	390	prg
Ethyl methacrylate	0.31	1	550	prg
Ethyl benzene	0.076	0.5	700	mcl
Methacrylonitrile	0.15	1	1	prg
Methyl methacrylate	0.29	2	1400	prg
Methylene chloride (dichloromethane)	0.19	3	4.3 ca	prg
Styrene	0.10	0.5	100	mcl
Tetrachloroethene	0.16	0.5	1.1 ca	prg
Toluene	0.04	0.5	720	prg
Total Xylenes	0.18	1	1400 (m&o)	prg
trans-1,2-Dichloroethene (trans-1,2-dichloroethylene)	0.10	0.5	100	[5]
trans-1,3-Dichloropropene	0.10	0.5	0.5[9] ca	[5]
trans-1,4-Dichloro-2-butene	0.34	10	10[10] ca	[5]
Trichloroethene	0.12	0.5	1.6 ca**	prg
Trichlorofluoromethane (fluorotrichloromethane)	0.14	0.5	1300	prg
Vinyl acetate	0.21	0.5	410	prg
Vinyl chloride	0.20	0.5	2 ca	[5]
2-Chloro-1,3-butadiene (chloroprene)	0.40	3	14	prg

ug/L micrograms per liter
* Asterisks indicate those chemicals for which the laboratory reporting limit (RL) exceeds the risk-based target level for the project.
1 Method detection limits (MDLs) (all parameters except metals), instrument detection limits (IDLs) (metals only), and reporting limits (RLs) as provided by Laucks Testing Laboratories, Inc. These values may change as laboratory MDLs and IDLs are updated.
2 Value is based on human health risk-based criteria or practical quantitation limits (PQLs) for common laboratory analytical methods.
3 Risk-based target level is not provided, because human health risk-based criteria are not available for this chemical.
4 Laucks Testing Laboratories is confident that it can reliably report to this PQL, even though this value is less than two times the MDL.
5 Developed using U.S. EPA Region 5 support. Risk-based target level numbers based on conversations with Crane (Paul Freed/Carol Witt-Smith fax dated 9/12/97). The value is based on human health criteria or practical quantitation limits (PQLs) for common analytical methods.
6 Being remanded.
7 Hexavalent chromium (Cr^{+6}) will be analyzed in the field using a Hach test kit, Method 8023 (see Appendix E fo the Work Plan). Powder pillows or AccuVac ampuls will be used. Detection limits for both methods are 10 ug/L.
8 1,4-Dioxane, acetonitrile, isobutyl alcohol, and propionitrile exluded from Appendix IX volatile target list because these compounds cannot be accurately analyzed using SW-846 Method 8260B, and they are not expected to be present at the Landfarm based on generator knowledge.
9 1,3-Dichloropropene
10 1,4-Dichloro-2-butene
prg - Region 9 preliminary remedial goal (PRG) for tap water, expiration 5/1/99.
mcl - Maximum contaminant level, Drinking Water and Health Advisories, October 1996.
smcl Secondary maximum contaminant level (for taste, odor, etc.)
ca Cancer PRG
ca ** (where: non-carcinogenic (nc) <100X ca)
ca *** (where: nc<10Xca)

- Reviewing past operations and integrating the information to develop data needs for the project.

- Developing a sampling and analysis plan that results in data representative of actual ground water conditions at the unit, including background.

- Selecting methods of analysis that are sensitive enough to meet the proposed risk-based target levels.

- Obtaining and handling representative ground water samples through standardized and documented sampling procedures.

- Statistically comparing the resulting analytical data to the proposed risk-based target levels and background.

1.5 SAMPLE NETWORK DESIGN AND RATIONALE

The sample network design and rationale is discussed in detail in Section 1.4 of the Work Plan. Figure 1-3, provided in Section 1.3 of the Work Plan, displays the locations of all monitoring wells.

1.6 PROJECT SCHEDULE

The project schedule is provided in Section 1.8 of the Work Plan.

2.0 PROJECT ORGANIZATION

The project organization for the SWMU 30 NSWC Crane RFI is provided in Section 1.5 of the Work Plan. A project organization chart and responsibilities of key personnel are provided.

3.0 QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA

The overall quality assurance (QA) objective for this project is to develop and implement procedures for field sampling, chain-of-custody, laboratory analysis, and reporting that will provide technically sound results. Intended data uses are described in Section 1.4.2 of this QAPP. Specific procedures for sampling, chain-of-custody, laboratory instrument calibration, laboratory analysis, data reporting, internal quality control (QC), audits, preventive maintenance of field and laboratory equipment, and corrective action are described in other sections of this QAPP.

The PARCC parameters (precision, accuracy, representativeness, comparability, and completeness) are qualitative or quantitative statements regarding the quality characteristics of the data used to support project objectives and ultimately, environmental decisions. These parameters are discussed in the remainder of this section. Specific routine procedures used to assess the quantitative parameters (precision, accuracy, and completeness) are provided in Section 12.0.

3.1 PRECISION

3.1.1 Definition

Precision is a measure of the amount of variability and bias inherent in a data set. Precision describes the reproducibility of measurements of the same parameter for samples under similar conditions. The equation for determining precision is provided in Section 12.2.

3.1.2 Field Precision Objectives

Field duplicate precision monitors the consistency with which environmental samples were obtained and analyzed. Field duplicate results for aqueous matrix samples are considered to be precise if the relative percent difference (RPD) is less than or equal to 30 percent. Field precision is assessed through the collection and measurement of field duplicates at a rate of 1 duplicate per 10 environmental samples.

3.1.3 Laboratory Precision Objectives

Laboratory precision QC samples are analyzed at a frequency of 5 percent (i.e., 1 QC sample per 20 environmental samples). Laboratory precision is measured by comparing calculated RPD values and precision control limits specified in the analytical method or by the laboratory's QA/QC program. Precision for organic analyses will be measured via RPDs for matrix spike and matrix spike duplicate

(MS/MSD) samples. Precision for metals and general chemistry analyses will be measured via RPDs for laboratory duplicates. Tables 3-1, 3-3, 3-5, and 3-7 present precision control limits for MS/MSD and laboratory duplicate RPDs, as applicable, for each analytical fraction. Tables 3-2, 3-4, 3-6, and 3-8 present accuracy control limits, which are discussed in Section 3.2.

3.2 ACCURACY

3.2.1 Definition

Accuracy is the degree of agreement between an observed value and an accepted reference value. The equation for determining accuracy is provided in Section 12.1.

3.2.2 Field Accuracy Objectives

Accuracy in the field is assessed through the use of rinsate and trip blanks and is ensured through the adherence to all sample handling, preservation, and holding time requirements. Accuracy and precision requirements for field measurements (e.g., pH) are ensured through calibration, as discussed in Section 2.4 of the Work Plan.

3.2.3 Laboratory Accuracy Objectives

Accuracy in the laboratory is measured by comparing a spiked sample result to a known or calculated value expressed as percent recovery (%R). Percent recoveries are derived from the analysis of known amounts of compounds spiked into deionized water [i.e., laboratory control sample (LCS) analysis], or into actual samples (i.e., surrogate or MS analysis). LCS analysis, which may also be referenced as blank spike analysis, measures the accuracy of laboratory operations. Surrogate and MS analyses measure the accuracy of laboratory operations as affected by sample matrix. LCS or MS analyses are performed at a frequency of 1 per 20 associated samples of like matrix. Surrogate spike analysis is performed for all organic chromatographic analyses. Laboratory accuracy is assessed by comparing calculated %R values to accuracy control limits specified in the analytical method or by the laboratory's QA/QC Program.

Accuracy for organic analyses will be measured via percent recoveries for surrogate spikes, MS/MSDs, and LCSs. Accuracy for metals and general chemistry analyses will be measured via percent recoveries for MSs and LCSs. Tables 3-2, 3-4, 3-6, and 3-8 present accuracy control limits.

TABLE 3-1

QUALITY CONTROL LIMITS[1]
MATRIX SPIKE/MATRIX SPIKE DUPLICATE SAMPLES AND SURROGATE SPIKES
EXPLOSIVES ANALYSES
NAVAL SURFACE WARFARE CENTER
CRANE, INDIANA

EXPLOSIVES BY SW-846 METHOD 8330

Chemical	Accuracy (%R)	Precision (RPD)
1,3,5-Trinitrobenzene	44-142	30
1,3-Dinitrobenzene	32-122	30
2,4,6-Trinitrotoluene	59-114	30
2,4-Dinitrotoluene	42-110	30
2,6-Dinitrotoluene	38-106	30
HMX	10-96	30
2-Nitrotoluene	30-99	30
3-Nitrotoluene	28-105	30
4-Nitrotoluene	31-100	30
4-Amino-2,6-dinitrotoluene	58-117	30
2-Amino-4,6-dinitrotoluene	58-117	30
Tetryl	27-109	30
Nitrobenzene	31-99	30
RDX	47-125	30
1,2-Dinitrobenzene (surrogate)	60-140	NA[2]

1 In-house QC limits provided by Laucks Testing Laboratories, Inc.
2 Not applicable
%R Percent recovery
RPD Relative percent difference

TABLE 3-2

QUALITY CONTROL LIMITS[1]
LABORATORY CONTROL SAMPLES
EXPLOSIVES ANALYSES
NAVAL SURFACE WARFARE CENTER
CRANE, INDIANA

EXPLOSIVES BY SW-846 METHOD 8330

Chemical	Accuracy (%R)
1,3,5-Trinitrobenzene	52-125
1,3-Dinitrobenzene	53-122
2,4,6-Trinitrotoluene	51-134
2,4-Dinitrotoluene	55-129
2,6-Dinitrotoluene	49-132
HMX	29-135
2-Nitrotoluene	41-140
3-Nitrotoluene	40-145
4-Nitrotoluene	39-142
4-Amino-2,6-dinitrotoluene	60-125
2-Amino-4,6-dinitrotoluene	61-125
Methyl-2,4,6-Trinitrophenylnitramine	33-138
Nitrobenzene	40-134
RDX	44-125

1 In-house QC limits provided by Laucks Testing Laboratories, Inc.
%R Percent recovery

TABLE 3-3

QUALITY CONTROL LIMITS[1]
MATRIX SPIKE AND LABORATORY DUPLICATE SAMPLES
METALS ANALYSES
NAVAL SURFACE WARFARE CENTER
CRANE, INDIANA

APPENDIX IX METALS BY SW-846 METHOD 6020 (UNLESS OTHERWISE NOTED)

Chemical	Accuracy (%R)	Precision (RPD)
Antimony	75-125	20
Arsenic	75-125	20
Barium	75-125	20
Beryllium	75-125	20
Cadmium	75-125	20
Chromium (total)	75-125	20
Cobalt	75-125	20
Copper	75-125	20
Lead	75-125	20
Mercury (SW-846 Method 7470A)	75-125	20
Nickel	75-125	20
Selenium	75-125	20
Silver	75-125	20
Thallium	75-125	20
Tin	75-125	20
Vanadium	75-125	20
Zinc	75-125	20

1 In-house QC limits provided by Laucks Testing Laboratories, Inc.

%R Percent recovery

RPD Relative percent difference

TABLE 3-4

QUALITY CONTROL LIMITS[1]
LABORATORY CONTROL SAMPLES
METALS ANALYSES
NAVAL SURFACE WARFARE CENTER
CRANE, INDIANA

APPENDIX IX METALS BY SW-846 METHOD 6020 (UNLESS OTHERWISE NOTED)

Chemical	Accuracy (%R)
Antimony	75-125
Arsenic	80-120
Barium	80-120
Beryllium	80-120
Cadmium	80-120
Chromium (total)	80-120
Cobalt	80-120
Copper	80-120
Lead	80-120
Mercury (SW-846 Method 7470A)	80-120
Nickel	80-120
Selenium	80-120
Silver	75-125
Thallium	80-120
Tin	50-150
Vanadium	80-120
Zinc	80-120

1 In-house QC limits provided by Laucks Testing Laboratories, Inc.

%R Percent recovery

TABLE 3-5

QUALITY CONTROL LIMITS[1]
MATRIX SPIKE/MATRIX SPIKE DUPLICATE SAMPLES AND SURROGATE SPIKES
VOLATILE ORGANIC COMPOUND ANALYSES
NAVAL SURFACE WARFARE CENTER
CRANE, INDIANA

VOLATILE ORGANIC COMPOUNDS BY SW-846 METHOD 8260B

Chemical	Accuracy (%R)	Precision (RPD)
1,1,1-Trichloroethane	75-125	20
1,1,2,2-Tetrachloroethane	74-125	20
1,1,2-Trichloroethane	75-127	20
1,1-Dichloroethane	72-125	20
1,1-Dichloroethene	59-145	20
1,2-Dichloroethane	68-127	20
1,2-Dichloropropane	70-125	20
2-Butanone	70-125	20
2-Hexanone	70-125	20
4-Methyl-2-pentanone	70-125	20
Acetone	70-125	20
Benzene	62-142	20
Bromodichloromethane	75-125	20
Bromoform	75-125	20
Bromomethane	72-175	20
Carbon disulfide	70-125	20
Carbon tetrachloride	62-125	20
Chlorobenzene	62-135	20
Chloroethane	65-125	20
Chloroform	74-125	20
Chloromethane	75-125	20
cis-1,2-Dichloroethene	75-125	20
trans-1,2-Dichloroethene	75-125	20
cis-1,3-Dichloropropene	74-125	20
trans-1,3-Dichloropropene	66-125	20
Dibromochloromethane	75-125	20
Ethylbenzene	75-125	20
Methylene chloride	75-125	20
Styrene	75-125	20
Tetrachloroethene	71-125	20
Toluene	59-139	20
Trichloroethene	54-141	20
Vinyl chloride	46-134	20
Xylenes (Total)	75-125	20
Toluene-D8 (surrogate)	75-125	NA[2]
1,2-Dichloroethane-D4 (surrogate)	62-139	NA
4-Bromofluorobenzene (surrogate)	75-125	NA

1 In-house QC limits provided by Laucks Testing Laboratories, Inc.
2 Not applicable.

%R Percent Recovery
RPD Relative percent difference

TABLE 3-6

QUALITY CONTROL LIMITS[1]
LABORATORY CONTROL SAMPLES
VOLATILE ORGANIC COMPOUND ANALYSES
NAVAL SURFACE WARFARE CENTER
CRANE, INDIANA

VOLATILE ORGANIC COMPOUNDS BY SW-846 METHOD 8260B

Chemical	Accuracy (%R)
1,1-Dichloroethene	71-130
Benzene	85-122
Chlorobenzene	84-114
Trichloroethene	86-117
Toluene	80-120

1 In-house QC limits provided by Laucks Testing Laboratories, Inc.

%R Percent recovery

TABLE 3-7

QUALITY CONTROL LIMITS[1]
MATRIX SPIKE AND LABORATORY DUPLICATE SAMPLES
MISCELLANEOUS PARAMETERS
NAVAL SURFACE WARFARE CENTER
CRANE, INDIANA

MISCELLANEOUS PARAMETERS

Chemical	Accuracy (%R)	Precision (RPD)
Ammonia (EPA Method 350.1)	53-120	10
Cyanide (SW-846 Method 9012A)	64-135	11
Nitrate/Nitrate (EPA method 353.2)	68-122	10
Phosphorus (EPA Method 365.2)	54-129	27
Total Kjeldahl Nitrogen (EPA Method 351.2)	50-150	30

1 In-house QC limits provided by Laucks Testing Laboratories, Inc.
2 Statistical QC limits will be developed once 20 data points are obtained. The default limits presented will be used until that time.
%R Percent Recovery
RPD Relative percent difference

TABLE 3-8

QUALITY CONTROL LIMITS[1]
LABORATORY CONTROL SAMPLES
MISCELLANEOUS PARAMETERS
NAVAL SURFACE WARFARE CENTER
CRANE, INDIANA

MISCELLANEOUS PARAMETERS

Chemical	Accuracy (%R)
Ammonia (EPA Method 350.1)	75-125
Cyanide (SW-846 Method 9012A)	75-125
Nitrate/Nitrate (EPA Method 353.2)	86-114
Phosphorus (EPA Method 365.2)	75-125
Total Kjeldahl Nitrogen (EPA method 351.2)	50-150

1 In-house QC limits provided by Laucks Testing Laboratories, Inc.
2 Statistical QC limits will be developed once 20 data points are obtained. The default limits presented will be used until that time.
%R Percent recovery

3.3 COMPLETENESS

3.3.1 Definition

Completeness is a measure of the amount of usable, valid analytical data obtained compared to the amount expected to be obtained. Completeness is typically expressed as a percentage. The equation for completeness is presented in Section 12.3.

The ideal objective for completeness is 100 percent (i.e., every sample planned to be collected is collected; every sample submitted for analysis yields valid data). However, samples can be rendered unusable during shipping or preparation (e.g., bottles broken or extracts accidentally destroyed), errors can be introduced during analysis (e.g., loss of instrument sensitivity, introduction of ambient laboratory contamination), or strong matrix effects can become apparent (e.g., extremely low matrix spike recovery). These instances result in data that do not meet QC criteria. Based on these considerations, 95 percent is considered an acceptable target for the data completeness objective. Completeness will be calculated for each quarterly sampling event of the ground water monitoring program. If critical data points are lost, resampling or reanalysis may be required.

Validation will be performed for 100 percent of the laboratory data for the ground water monitoring program based on the requirements of the analytical methods and this QAPP. To the extent practicable for SW-846 analysis, validation will also be performed in accordance with the Region 5 standard operating procedures (SOPs) for Validation of CLP (Contract Laboratory Program) Organic and Inorganic Data (U.S. EPA Region 5, 1993a, 1993b) and the U.S. EPA Contract Laboratory Program National Functional Guidelines for Organic and Inorganic Data Review (U.S. EPA, 1994a, 1994b). Data rejected as a result of the validation process will be treated as unreliable, unusable data.

3.3.2 Field Completeness Objectives

Field completeness is a measure of the amount of valid field measurements obtained from all the field measurements taken in the project. Field completeness for this project is expected to be greater than 95 percent.

3.3.3 Laboratory Completeness Objectives

Laboratory completeness is a measure of the amount of valid laboratory measurements obtained from all the laboratory measurements taken in the project. Laboratory completeness for this project is expected to be at least 95 percent.

3.4 REPRESENTATIVENESS

3.4.1 Definition

Representativeness is an expression of the degree to which the data accurately and precisely depict the actual characteristics of a population or environmental condition at an individual sampling point. Use of standardized sampling, handling, analytical, and reporting procedures ensures that the final data accurately represent actual site conditions.

3.4.2 Measures to Ensure Representativeness of Field Data

Representativeness is dependent on the proper design of the sampling program and will be satisfied by ensuring that the Work Plan is followed and that proper sampling techniques are used.

3.4.3 Measures to Ensure Representativeness of Laboratory Data

Representativeness in the laboratory is ensured by using the proper analytical procedures, meeting sample holding times, and analyzing and assessing field duplicate samples. The sampling network for the NSWC Crane RFI was designed to provide data representative of facility conditions. During development of this network, consideration was given to past waste disposal practices, physical setting and processes, and constraints inherent to the RCRA program. The rationale of the sampling network is discussed in detail in Section 4 of the Work Plan.

3.5 COMPARABILITY

3.5.1 Definition

Comparability is defined as the confidence with which one data set can be compared to another (e.g., between sampling points; between sampling events). Comparability is achieved by using standardized sampling and analysis methods and data reporting formats (including use of consistent units of measure). Additionally, consideration is given to seasonal conditions and other environmental variations that could influence data results.

3.5.2 Measures to Ensure Comparability of Field Data

Comparability is dependent on the proper design of the sampling program and will be satisfied by ensuring that the Work Plan is followed and that proper sampling techniques are used. The sample network design and rationale is presented in Section 1.4 of the Work Plan. Comparability is also dependent on recording field measurements using the correct units. Field measurement units are further discussed in Section 9.1.1.

3.5.3 Measures to Ensure Comparability of Laboratory Data

Planned analytical data are comparable when similar sampling and analytical methods are used and documented. Results will be reported in units that ensure comparability with current state and federal standards and guidelines. Laboratory measurement units are further discussed in Section 9.1.2.

3.6 LEVEL OF QUALITY CONTROL EFFORT

Trip blank, rinsate blank, source water blank, field duplicate, method blank, laboratory duplicate, laboratory control, and MS samples will be analyzed to assess the quality of the data resulting from the field sampling and analytical programs. Internal QC samples (i.e., laboratory QC samples) are addressed in Section 8.0 of this QAPP.

External QC measures (i.e., field QC samples) consist of field duplicates, trip blanks, and equipment rinsate blanks. Information gained from these analyses further characterizes the level of data quality obtained to support project goals. Each of these types of field QC samples undergo the same preservation, analysis, and reporting procedures as the related environmental samples. Each type of field QC sample is discussed below.

In terms of ground water sampling, field duplicates for ground water samples are two samples collected independently at the source sampling location and analyzed for the same parameters. Field duplicates are collected and analyzed for chemical constituents to measure the precision of the sampling and analysis methods employed. Field duplicates will also be collected and analyzed for field parameters to be performed using field test kits (i.e., hexavalent chromium). The general level of the QC effort will be 1 field duplicate for every 10 or fewer investigative samples.

Trip blanks will be submitted to the laboratory to provide a means to assess the quality of the data resulting from the field sampling program.

Trip blanks pertain to samples collected for VOC analysis only. Trip blanks are used to assess the potential for VOC contamination resulting from contaminant migration into sample containers during sample shipment and storage. Trip blanks are prepared by the laboratory using organic-free reagent water prior to the sampling event. They are shipped to the site with the sample containers and kept with the investigative samples throughout the sampling event. They are then packaged for shipment with other VOC samples and sent to the laboratory for analysis. One trip blank will be included in each sample shipping container that contains samples for VOC analysis. At no time after trip blank preparation will the trip blank sample containers be opened before they reach the laboratory.

Equipment rinsate blanks are used to assess the effectiveness of decontamination procedures. They are obtained under representative field conditions by collecting the rinse water generated by running analyte-free water through sample collection equipment after decontamination and prior to use. One rinsate blank per ten environmental samples will be collected per each type of sampling equipment used (i.e., bailer, hand tools, etc.). However, at least one equipment blank will be collected per sampling day, per sampling event. If pre-cleaned, dedicated, or disposable sampling equipment is used, one rinsate blank must be collected as a "batch blank." Rinsate blanks are analyzed for the same chemical constituents as the associated environmental samples.

Source water blanks are obtained by sampling the waters used for decontamination during the field investigation. Samples consist of analyte-free water used for decontaminating sampling equipment. Source water blanks will be used to determine if the analyte-free water or the laboratory bottles are contributing to sample contamination. Source water blanks will be collected for each type of water used for decontamination and will be submitted at a frequency of one per sampling event. Source water blanks, as applicable, will be analyzed for the entire suite of parameters under investigation. It is anticipated that one source water blank will be collected for each sampling round.

Laboratory duplicate samples are analyzed for inorganic parameters to check for sampling and analytical reproducibility. MSs are investigative samples analyzed to provide information about the effect of the sample matrix on the preparation and measurement methodology. All MSs for organic analyses are performed in duplicate and, as previously defined, are referred to as MS/MSD samples. One MS/laboratory duplicate or MS/MSD sample set will be collected or designated for every 20 or fewer investigative samples. Extra sample volume must be collected for samples designated for MS/MSD analysis for VOCs and explosives. Specific details regarding extra sample volume required for MS/MSD samples are provided in Table 2-3 of the Work Plan for each applicable analytical fraction. MS/MSD samples are further discussed in Section 8.0.

Table 2-1 of the Work Plan indicates the total number of each QC sample to be collected on the basis of parameter and sampling event.

4.0 SAMPLING PROCEDURES

Field sampling procedures and additional field investigation tasks for the NSWC Crane SWMU 30 RFI are discussed in detail in the following sections of the Work Plan.

Section	Topic
2.3	GROUND WATER SAMPLING
2.3.1	Water-Level Measurements and Ground Water Monitoring Well Evaluations
2.3.2	Monitoring Well Development and Purging
2.3.3	Sampling
2.4	FIELD MEASUREMENTS
2.5	DECONTAMINATION
2.6	INVESTIGATION DERIVED WASTE (IDW)
3.1	SAMPLING IDENTIFICATION SYSTEM
3.2	SAMPLE PACKAGING AND SHIPPING
3.3	SAMPLE CUSTODY
3.4	QUALITY CONTROL SAMPLES
3.5	RECORDKEEPING

Standard Operating Procedures (SOPs) regarding sampling and recordkeeping are included as Appendix C of the Work Plan.

5.0 CUSTODY PROCEDURES

Custody is one of several factors necessary for the admissibility of environmental data as evidence in a court of law. Custody procedures help to satisfy the two major requirements for admissibility: relevance and authenticity. Sample custody is addressed in three parts: field sample collection, laboratory analysis, and final evidence files. Final evidence files, including all originals of laboratory reports, are maintained under document control in a secure area. A sample or evidence file is under custody if:

- the item is in the actual physical possession of an authorized person, or
- the item is in view of the person after being in his or her possession, or
- the item was placed in a secure area to prevent tampering, or
- the item is in a designated and identified secure area with access restricted to authorized personnel only.

The chain-of-custody (COC) report is a multi-part, standardized form used to summarize and document pertinent sample information such as sample identification and type, matrix, date and time of collection, preservation, and requested analyses. Furthermore, through the sequential signatures of various sample custodians (e.g., sampler, transporter, laboratory sample custodian), the COC report documents sample custody and tracking. Custody procedures apply to all environmental and associated field QC samples obtained as part of the data collection system.

5.1 FIELD CUSTODY PROCEDURES

The Field Operations Leader (FOL) or designee is responsible for the care and custody of the samples collected until they are relinquished to the analyzing laboratory or entrusted to a commercial overnight courier. COC reports are completed for each sample shipment. The reports are filled out in a legible manner, using waterproof ink, and are signed and dated by the sampler. Pertinent notes, such as whether the sample was field-filtered, or whether the sample is suspected to be high in contaminant concentration, are also indicated on the COC report. Information similar to that contained in the COC report is also provided on the sample label, which is securely attached to the sample bottle. In addition, sample tags are affixed to the sample bottles and are returned by the analytical laboratory for inclusion in the final evidence file.

Full details regarding sample COC (including use of custody seals and sample shipment protocols) are contained in SOP SA-6.1, which is provided in Appendix C of the Work Plan. SOP SA-6.3, also provided in Appendix C of the Work Plan, discusses maintenance of site logbooks, site notebooks, and other field

records. Additionally, each of the sampling SOPs incorporated into the Work Plan contains a section that addresses relevant sample documentation (completion of sample logsheets, etc.). All sample records are eventually docketed into the Administrative Record at NSWC Crane.

5.2 LABORATORY CUSTODY PROCEDURES

When samples are received by the laboratory subcontractor, the laboratory's sample custodian examines the custody seals on each cooler to verify that they are intact and that the integrity of the environmental samples has been maintained. The custodian then opens the cooler and measures its internal temperature by measuring the temperature of the temperature blank. (A temperature blank is included in each cooler.) The temperature reading is documented on the accompanying COC report. The sample custodian then signs the COC report and examines the contents of the cooler. Sample container breakages or discrepancies between the COC report and sample label documentation are recorded. With the exception of samples for VOC analysis, the pH of chemically preserved samples is checked using Hydrion paper and recorded. (The pH of VOC samples will be measured and recorded after analysis to prevent loss of volatile compounds.) The laboratory completes a Laucks Testing Laboratories, Inc., CLP Sample Receipt Log and Supplemental Sample Receipt Log, as shown in Appendix 3 of SOP LTL-4002 (Appendix A). All problems or discrepancies noted during this process are to be promptly reported to the TtNUS Task Order Manager (TOM). Samples are then logged into the laboratory information management system (LIMS). Other pertinent issues related to sample custody, such as inter-laboratory chain-of-custody procedures and specific procedures for sample handling, storage, dispersement for analysis, and remnant disposal, are discussed in the subcontracted laboratory SOPs included in Appendix A of this QAPP.

5.3 FINAL EVIDENCE FILES

The Administrative Record at NSWC Crane will be the repository for all documents that constitute evidence relevant to sampling and analysis activities described in this QAPP. NSWC Crane will be the custodian of the evidence files and will maintain the contents of these files for the SWMU 30 RFI, including all relevant records, reports, logs, field notebooks, photographs, subcontractor reports, and data reviews, in a secure, limited access location and under custody of the NSWC Installation Restoration Project Manager. The control file will include at a minimum:

- field logbooks
- field data and data deliverables
- photographs and negatives

- drawings
- laboratory data deliverables
- data validation reports
- data assessment reports
- progress reports, QA reports, interim project reports, etc.
- all custody documentation (tags, chain-of-custody forms, airbills, etc.)

Upon completion of the contract, all files associated with the ground water program will be maintained in the Administrative Record at NSWC Crane and will be available for inspection by the regulatory agencies. Section 9.3.2 provides additional details regarding laboratory data deliverables and reporting.

6.0 CALIBRATION PROCEDURES AND FREQUENCY

All instrumentation used to perform chemical measurements must be properly calibrated prior to use in order to obtain valid and usable results. The requirement to properly calibrate instruments prior to use applies equally to field instruments and fixed-base laboratory instruments. Field instrument calibration is discussed in Section 6.1. Laboratory instrument calibration is discussed in Section 6.2.

6.1 FIELD INSTRUMENT CALIBRATION

Field instrument calibration is discussed in Section 2.4 of the Work Plan.

6.2 LABORATORY INSTRUMENT CALIBRATION

Calibration procedures for a specific laboratory instrument will consist of initial calibration (generally three to five points), initial calibration verification (inorganic methods only), and continuing calibration verification. In all cases, an independently prepared standard (i.e., from a second source or a different lot number from the primary source) will be used as a calibration verification solution or as the LCS/MS spiking mix.

All standards used to calibrate analytical instruments must be obtained from the National Institute of Standards and Technology (NIST) or through a reliable commercial supplier with a proven record for quality standards. All commercially supplied standards must be traceable to NIST reference standards, where possible, and appropriate documentation will be obtained from the supplier. In cases where documentation is not available, the laboratory will analyze the standard and compare the results to an U.S. EPA-supplied known or previous NIST-traceable standard.

Laboratory instrumentation calibration procedures, frequency requirements, acceptance criteria, and corrective action are described in the method-specific SOPs presented in Appendix A of this QAPP.

7.0 ANALYTICAL AND MEASUREMENT PROCEDURES

All ground water samples collected during the NSWC Crane SWMU 30 RFI will be analyzed by Laucks Testing Laboratories, Inc. Laucks Testing Laboratories, Inc., is located at 940 South Harney Street, Seattle, Washington 98108, (206) 767-5060; FAX (206) 767-5063. This laboratory has successfully completed the Naval Facilities Engineering Service Center NFESC laboratory evaluation process described in the "Navy Installation Restoration Laboratory Quality Assurance Guide" (NFESC, February 1996).

Samples will be subjected to field parameter measurement and will be analyzed at the fixed-base laboratory for volatiles, metals (total and dissolved), ammonia, cyanide, hexavalent chromium, nitrate/nitrite, phosphorus (total and total dissolved), and TKN. As described in Section 1.4.2.2, the parameter list may vary based on sampling round.

The remainder of this section discusses field measurements and analytical procedures in more detail.

7.1 FIELD MEASUREMENT PROCEDURES

Chemical and physical parameters to be measured using field instrumentation include volatile organics as methane equivalents (air quality within well casings), dissolved oxygen, temperature, specific conductivity, oxidation reduction potential, pH, turbidity, and water level. Hexavalent chromium (Cr^{+6}) will also be analyzed in the field using the Hach test kit, method 8023 (see Appendix E of the Work Plan). AccuVac ampuls will be used with a detection limit of 10 µg/L (Cr^{+6} criterion is 180 µg/L). Measurement of field parameters and calibration of field instruments are discussed in Section 2.4 (Field Measurements) of the Work Plan.

7.2 LABORATORY ANALYTICAL AND MEASUREMENT PROCEDURES

All samples will be analyzed in accordance with the laboratory SOPs included in Appendix A of this QAPP. Table 7-1 summarizes the laboratory analytical methods and associated SOPs for the NSWC Crane SWMU 30 RFI.

TABLE 7-1

LABORATORY
SUMMARY OF ANALYTICAL PROCEDURES
NSWC CRANE, INDIANA

Analytical Parameter	Preparation Method	Analytical Method	Preparation/Analytical SOPs(1)
Appendix IX Volatile Organics(2)	SW-846(3) 5030A	SW-846(2) 8260B(with 25 mL purge)	LTL-8265(low-level option)
Explosives	SW-846 8330	SW-846 8330	LTL-3077/LTL-8330
Mercury, total and dissolved	SW-846 7470A	SW-846 7470A	LTL-7501
Appendix IX Metals (except mercury), dissolved	(5)	SW-846 6020	LTL-7202
Appendix IX Metals (except mercury), Total	SW-846 3010A or SW-846 3015	SW-846 6020	LTL-7009 or LTL-7010/LTL-7202
Sulfides	SW-846 9030B		LTL-9205
Nitrate/Nitrite	(5)	EPA(4) 353.2	LTL-9125
Ammonia	(5)	EPA 350.1	LTL-9109
Phosphorus, total and dissolved	(5)	EPA 365.2	LTL-9108
Total Kjeldahl Nitrogen	(5)	EPA 351.2	LTL-9133
Cyanide	(5)	SW-846 9012A	LTL-9104

1 All laboratory SOPs are included in Appendix A of this QAPP.
2 1,4-Dioxane, acetonitrile, isobutyl alcohol, and propionitrile are excluded from Appendix IX volatiles list because these compounds cannot be accurately analyzed using SW-846 method 8260B, and they are not expected to be present at the Landfarm based on general knowledge.
3 U.S. EPA, 1986b.
4 U.S. EPA, 1983 and U.S. EPA, 1993.
5 No preparation method is required.
mL milliliter

7.2.1 List of Project Target Compounds and Detection Limits

A complete list of the target compounds/analytes, including project-specific risk-based target level RLs, MDLs, and IDLs, is provided in Section 1.4.2.2 of this QAPP. The MDLs shown have been experimentally determined using the procedures described in Section 6.3 of Laucks Testing Laboratories, Inc., SOP LTL-1011, which is included in Appendix A of this QAPP. These procedures are based on the method provided in Federal Register Vol. 49, No. 209, pages 198-199 (40 CFR Part 136 Appendix B). The IDLs provided for metals have been experimentally determined as described in Section 6.2.6 of SOP LTL-1011. This procedure is based on the procedure for IDL determination as specified in the CLP Statement of Work (ILM04.0, U.S. EPA, 1995). All analytical results will be reported to the analyte's laboratory-specific RL as specified in Table 1-2 or to the lowest calibration standard used for that analyte. An analyte's RL is laboratory-specific and is based on the associated MDL/IDL, with adjustments made to ensure that the precision and accuracy requirements of the method are attainable. RLs will be adjusted on a sample-by-sample basis, as necessary, based on dilutions and sample volume. These adjusted values are known as sample quantitation limits (SQLs).

7.2.2 List of Associated Quality Control Samples

In addition to the field QC samples (field duplicates, trip blanks, rinsate blanks, etc.) discussed in Section 3.0 of this QAPP, laboratory QC samples including MS/MSD samples, method blanks, and preparation blanks, will be analyzed as required by the analytical methods and as stated in the laboratory SOPs included in Appendix A of this QAPP. Laboratory QC samples are further discussed in Section 8.0 of this QAPP.

8.0 INTERNAL QUALITY CONTROL CHECKS

Field-related QC checks are discussed in Section 3.0 of this QAPP. This section provides additional information regarding internal QC checks for field and laboratory measurements and analyses.

8.1 FIELD QUALITY CONTROL CHECKS

Internal QC procedures for pH, specific conductivity, temperature, dissolved oxygen, ORP, and turbidity will include calibrating the instruments as described in Section 2.4 of the Work Plan and in SOP SA-1.1 provided in Appendix C of the Work Plan. Internal QC procedures for hexavalent chromium are described in the HACH procedure included in Appendix E of the Work Plan. Field sampling precision and bias will be assessed by collecting field duplicates and rinsate blanks for laboratory analysis. Collection of QC samples is discussed in Sections 2.3.3 and 2.4 of the Work Plan. Collection frequencies are summarized in Table 2-1 of the Work Plan.

8.2 LABORATORY QUALITY CONTROL CHECKS

The identified subcontract laboratory (Laucks Testing Laboratories, Inc.) has a QC program that ensures the reliability and validity of the analyses performed at the laboratory. All analytical procedures are documented in writing as SOPs. Internal QC procedures for all analyses are specified in the method-specific SOPs provided in Appendix A of this QAPP.

Table 7-1 lists the SOPs associated with each analytical procedure. In addition, the laboratory maintains SOPs regarding general laboratory QA procedures. All applicable SOPs, are also included in Appendix A. The table of contents included in Appendix A lists titles and numbers for all SOPs contained in the appendix. Several internal laboratory QC checks are briefly discussed in the remainder of this section. Additional QC requirements that are specific to the NFESC QA Program, and therefore required for this project, are also specified, as applicable, for each of the QC checks.

Laboratory method blanks are prepared and analyzed in accordance with the analytical method employed to determine whether contaminants originating from laboratory sources have been introduced and have affected environmental sample analyses. Method blanks for analytical methods that include preparative extraction or digestion procedures are also called preparation blanks. A method blank for ground water sample analysis generally consists of an aliquot of analyte-free water that is subjected to the same preparation and analysis procedures as the environmental samples undergoing analysis. Criteria for method blanks and corrective actions for noncompliant results are described in each SOP for

determinative analysis in Appendix A of this QAPP. Under no circumstances are laboratory method blank contaminant values subtracted from environmental sample analysis results.

Matrix spike analysis for organic analyses is performed in duplicate (i.e., MS/MSD analysis), with a frequency of 1 per 20 environmental samples as a measure of laboratory precision. For inorganic analyses, MS and laboratory duplicate analysis is likewise performed for every 20 environmental sample analyses. With the exception of VOC MSD analyses, laboratory duplicates are prepared by obtaining two sample aliquots from the same sample container and analyzing each portion following the same analytical procedures. For VOC MSD analyses, a sample aliquot from a separate sample container is used for analysis. The field crew provides extra volumes of sample matrices designated for laboratory QC analyses, as required. Control limits for matrix spike and laboratory duplicate analyses are specified in Section 3.0 of this QAPP.

Based on NFESC requirements, MS samples should contain all the targeted analytes of interest. However, because of the extensive list of compounds included on the Appendix IX list for VOCs and the overlapping retention times of some of these compounds, it is not feasible to spike and analyze for the full list of Appendix IX compounds in the MS/MSD samples. Therefore, MS/MSD samples for these VOCs will be spiked with a representative subset of these chemicals. Tables specifying matrix spiking compounds per analytical method and associated statistical laboratory control limits to be used for the ground water monitoring program are provided in Section 3.

If the MS recovery is not within applicable control limits, the laboratory will assess the batch to determine whether the spike results are attributable to a matrix effect or are the result of other problems in the analytical process. Based on NFESC requirements, if all the batch QC elements that are not affected by the sample matrix are in control (e.g., method blank, LCS, calibration checks) and if there is no evidence that spiking was improperly performed, the poor spike recovery may be attributed to matrix effects. In this case, the failed MS will be discussed in the laboratory case narrative, but re-preparation and re-analysis are not required. If any of the batch QC elements that are not affected by the sample matrix are out of control, or if there is evidence that spiking may have been improperly performed, the MS and MSD sample will be re-processed through the entire analytical sequence unless insufficient sample volume is available. Noncompliant MS/MSD and laboratory duplicate results will be discussed in the laboratory case narrative.

Surrogates are organic compounds (typically brominated, fluorinated, or isotopically labeled) that are similar in nature to the compounds of concern and that are not likely to be present in environmental media. Surrogates are spiked into each sample, standard, and method blank prior to analysis, and are

used only in organic chromatographic analysis procedures as a check of method effectiveness. Surrogate recoveries will be evaluated against the control limits specified in Section 3 of this QAPP. Corrective actions for noncompliant surrogate recoveries are discussed in the relevant determinative SOPs included in Appendix A of this QAPP. Noncompliant surrogate recoveries will be discussed in the laboratory case narrative.

Laboratory control samples or blank spike samples serve to monitor the overall performance of each step during the analysis, including sample preparation. LCSs must be included in each preparation or analytical batch of 20 samples or less, and must be analyzed utilizing the same sample preparations, analytical methods, and QA/QC procedures as those employed for the samples. Based on the requirements of the NFESC QA Program, LCSs for wet chemistry and metals analyses must contain all analytes of interest, whereas LCSs for multiple-analyte organic methods must contain at least two targeted analytes from each major class of compounds subject to analysis. The complete analyte list of explosives will be included in the LCSs associated with explosives analysis. The LCS spiking list for volatiles analyses will contain analytes that represent each of the various classes of analytes on the target analyte list. LCS results will be evaluated against control limits statistically established by the laboratory. Tables specifying LCS spiking compounds per analytical method and associated statistical laboratory control limits to be used for the NSWC Crane SWMU 30 RFI are provided in Section 3.0.

Based on NFESC QA Program requirements, if recovery of a LCS falls outside the control limits, the laboratory will reject the data for the analytical batch and take corrective action. The associated samples, extracts, or digestates may be reanalyzed a single time, and if the LCS recoveries meet acceptance criteria, the data will be reported. If LCS analyte recovery is still outside the acceptance limits, the associated samples in the preparation batch will be reprocessed if sufficient sample volume is available and holding times have not elapsed. If repreparation or reanalysis is not possible, the data will be flagged and the laboratory case narrative will discuss the failed LCS.

Internal standard performance criteria ensure that gas chromatography/mass spectrometry (GC/MS) analysis sensitivity and response are stable during every analytical run. Internal standard area counts for samples and blanks must not vary by more than a factor of two (- 50% to + 100%) from the associated 12-hour calibration standard. The retention time of the internal standards in samples and blanks must not vary by more than ±30 seconds from the retention time of the associated 12-hour calibration standard.

Additional internal laboratory QC checks include mass tuning for GC/MS VOC analysis, and second-column confirmation for high-performance liquid chromatography (HPLC) analyses. Specific QC requirements for each of these QC checks are provided in the applicable SOPs included in Appendix A of this QAPP.

9.0 DATA REDUCTION, VALIDATION, AND REPORTING

This section describes the procedures to be used for data reduction, validation, and reporting for the NSWC Crane SWMU 30 RFI. All data generated during the course of the RFI will be maintained in hard copy form in the Administrative Record at NSWC Crane.

9.1 DATA REDUCTION

Data reduction will be completed for both field measurements and laboratory-generated analytical data. Field data reduction will be relatively limited versus the degree of laboratory data reduction required for the project. Reduction of both field data and laboratory data are discussed in the remainder of this section.

9.1.1 Field Data Reduction

Field data will be generated as a result of real-time measurement of organic vapor concentrations via a photoionization detector (for health and safety monitoring) and through on-site water quality testing for general indicator parameters including pH, specific conductivity, turbidity, dissolved oxygen, ORP, and temperature. No calculations will be necessary to reduce these data. Measurements of hexavalent chromium will be performed using HACH test kits. Data generation for this parameter is described in the HACH procedure included in Appendix E of the Work Plan. No calculations will be necessary to reduce these data.

Field measurements of organic vapor concentrations (parts per million on a volume/volume basis relative to methane or benzene) will be recorded on sample logsheets immediately after measurements are taken; the results will be incorporated into the SWMU 30 RFI Report. General water quality indicator parameters will also be recorded on sample logsheets immediately after the measurements are taken and later encoded in the NSWC Crane SWMU 30 RFI database for presentation in the SWMU 30 Ground Water Assessment Report. If an error is made in the field records, the error will be legibly crossed out (single-line strikeout), initialed and dated by the field member, and corrected in a space adjacent to the original (erroneous) entry. Additional information regarding field documentation is provided in Section 3.5 of the Work Plan. With the possible exception of aquifer-related slug test calculations, no calculations will be necessary to reduce these data for inclusion in the SWMU 30 Ground Water Assessment Report. Field data will be entered in the electronic database manually, and the entries will be verified by an independent reviewer to make sure that no transcription errors occurred. General ground water quality data and field screening data will be recorded and reported in the following units:

- pH - standard pH units
- Temperature - degrees Celsius
- Specific Conductivity - milli-siemens per centimeter (mS/cm)
- Turbidity - Nephelometric turbidity units (NTU)
- Dissolved oxygen - milligrams per liter (mg/L)
- Oxidation Reduction Potential (ORP) - millivolts (mV)
- Water level - feet (ft)

9.1.2 Laboratory Data Reduction

Laboratory data reduction will be completed in accordance with the method-specific laboratory SOPs included in Appendix A of this QAPP. In addition, SOP LTL-1081 (provided in Appendix A) presents the procedures used by Laucks Testing Laboratories, Inc., for review and approval of data.

It should be noted that the potential for coelution exists for some of the explosive compounds. The list of explosive compounds that coelute may vary over time as new columns are installed or as column affinities change over time. [The use of a liquid phase as an eluent for high-performance liquid chromatography (HPLC) provides a likely chance for chemical changes to occur in the column substrate and/or active sites.]

SW-846 Method 8330 specifies that quantitation for explosive compounds be performed using the primary column. However, if the peaks for two compounds are observed to coelute on the primary column but the peaks for these same two compounds do not coelute on the confirmation column, results for these compounds will be quantitated and reported from the confirmation column. The laboratory will document this in the case narrative.

If two compounds coelute on the confirmation column, but the same two compounds do not coelute on the primary column, two scenarios could result. Quantitation will be performed using the primary column. Therefore, if only one of the coeluting compounds is detected on the primary column [and not the compound(s) it coelutes with], coelution on the confirmation column has no impact. The only potential for uncertainty is in the unlikely case that more than one of the coeluting compounds is detected on the primary column and a peak is also present in the expected retention time window on the confirmation column. In this case, it would not be possible to determine which compound(s) were actually present. Therefore, as a conservative measure, both compounds will be assumed to be present, and both will be quantitated and reported from the primary column. If this occurs, the results for these compounds will be

reported by the laboratory with a "Z" flag to indicate coelution has occurred on the confirmation column and that quantitative confirmation is not possible.

Laboratory analytical data will be reported using standard concentration units to ensure comparability with regulatory standards and guidelines and previous analytical results. Reporting units for the various classes of chemicals under consideration are as follows:

- Volatile organic compounds - µg/L
- Explosives - µg/L
- Hexavalent chromium – mg/L
- Metals - µg/L
- Cyanide - mg/L
- Nitrate/nitrite - mg/L
- Ammonia - mg/L
- Phosphorus - mg/L
- TKN - mg/L

9.2 DATA VALIDATION

This section discusses validation of field measurements and laboratory analytical data. Validation of field data will be limited to real-time "reality" checks, whereas laboratory analytical data will be validated in accordance with current U.S. EPA guidance. Validation of field measurements is discussed in Section 9.2.1. Validation of laboratory analytical data is discussed in Section 9.2.2.

9.2.1 Field Measurement Data Validation

Field measurements will not be subjected to a formal data validation process. However, field technicians will ensure that the equipment used for field measurement is performing accurately in compliance with the applicable SOPs. As described in Section 9.1.1, all field data entered into the electronic database will be independently reviewed for transcription errors.

9.2.2 Laboratory Data Validation

One hundred percent of the laboratory data will be validated to ensure that the data are of evidentiary quality. Validation of analytical data will be completed by the TtNUS Chemistry Department located in the

TtNUS Pittsburgh office. Final review and approval of validation deliverables will be completed by the TtNUS Data Validation Coordinator.

The analytical results will be validated versus the applicable analytical methods, the analytical SOPs included in Appendix A, and the requirements of this QAPP. Validation of these data will conform to the Region 5 Standard Operating Procedures for Validation of CLP Organic and Inorganic Data (U.S. EPA Region 5, 1993a, 1993b) and the National Functional Guidelines for Organic and Inorganic Data Review (U.S. EPA, 1994a, 1994b) to the greatest extent practicable for non-CLP data. The control limits provided in Tables 3-1 through 3-9 of this QAPP will be used to evaluate QC sample results.

As presented in Section 9.1.2 of the QAPP, the potential for coelution exists for some explosive compounds. Section 9.1.2 indicated that if more than one of the coeluting compounds is detected on the primary column and a peak is also present in the expected retention time window on the confirmation column, as a conservative measure, both compounds will be quantitated and reported by the laboratory from the primary column, and that the laboratory will qualify the results with a "Z" flag. If this occurs, the results for these compounds will be qualified as presumptively present at an estimated concentration ("JN") during data validation (replacing the "Z" flag applied by the laboratory).

9.3 DATA REPORTING

This section discusses data reporting requirements for field and laboratory analytical data. Section 9.3.1 discusses field measurement data handling and reporting. Section 9.3.2 discusses laboratory data handling and reporting.

9.3.1 Field Measurement Data Reporting

Field data will be reported in the units discussed in Section 9.1.1. The SWMU 30 Ground Water Assessment Report will include a comprehensive database including all field measurements (hexavalent chromium, pH, specific conductivity, temperature, dissolved oxygen, and turbidity). Field measurements will be transferred from sample logsheets to the electronic database manually and will be reviewed for accuracy by an independent reviewer. The addition of field measurements to the electronic database will be completed shortly after completion of the field investigation.

All records regarding field measurements (i.e., field logbooks, sampling logbooks, and sample logsheets) will be placed in the TtNUS central files upon completion of the field effort. Entry of these results in the database will require removal of these results from the files. Outcards will be used to document the

removal of any such documentation from the files (date, person, subject matter). Field measurement data will be reported in an appendix to the SWMU 30 Ground Water Assessment Report at a minimum, and may also be reported in summary fashion if they indicate the presence of contamination (e.g., high specific conductivity readings).

9.3.2 Laboratory Data Reporting

Data reporting by the laboratory will be in accordance with CLP reporting format to the extent practicable. All pertinent QC data including raw data and summary forms for blanks, surrogates, and calibration information, will be provided. Case narratives will be provided for each Sample Delivery Group (SDG). SOP LTL-4201 (Appendix A) provides further detail regarding the information that will be included in CLP-type data packages.

All environmental and field QC sample results (trip blanks, field duplicates, rinsate blanks) will be included in the SWMU 30 Ground Water Assessment Report as an appendix. The database will include pertinent sampling information such as sample number, monitoring well, and sampling date. Sample-specific detection limits will be reported for nondetected analytes. Units will be clearly summarized in the database and will conform to those identified in Section 9.1.2. The results for laboratory QC samples such as method blanks will not be presented in the RFI Report database. In addition, only the original (unspiked) sample results for MS/MSD samples will be included in the database.

The analytical data will also be summarized within the body of the SWMU 30 Ground Water Assessment Report text in tabular and graphic fashion. Tabular summaries will include only those analytes that are detected in at least one sample and may include information such as frequency of detection, mean concentration, and concentration range. In the event that graphical portrayals of data are informative, "tag maps," including the location and concentration of specific chemicals of potential concern, will be provided in the SWMU 30 Ground Water Assessment Report.

Data will be handled electronically pursuant to the electronic deliverable requirements specified in the TtNUS Basic Ordering Agreement with Laucks Testing Laboratories. This agreement requires the analytical laboratory to provide data in both hard copy and electronic form. The original electronic diskettes and the original hard copy analytical data are maintained in the Administrative Record at NSWC Crane. SOP CT-05 discusses database management and QA; it is included in Appendix A of this QAPP.

Validation will be completed using the hard copy data. Upon completion of validation of a SDG and review by the Data Validation Coordinator, the validation qualifiers will be entered in the electronic

database and will be subjected to independent review for accuracy. During this review process, the electronic database printout will also be compared to the hard copy data (Form Is) to ensure that the hard copy data and electronic data are consistent.

TtNUS will be responsible for laboratory data reporting. Key data handling personnel include the Information Management Systems Group leader and the Data Validation Coordinator. Data validation deliverables will not be routinely provided to U.S. EPA Region 5. However, a summary of the validation results (actions taken and completeness, precision, and accuracy) will be provided in the SWMU 30 Ground Water Assessment Report, and example data validation memoranda and analytical data packages will be provided upon request.

Statistical analysis of the analytical data will be performed as described in Section 1.4 of the Work Plan.

10.0 PERFORMANCE AND SYSTEM AUDITS

Performance and system audits will be conducted periodically to ensure that work is being implemented in accordance with the approved project plans and in an overall satisfactory manner. Examples of pertinent audits are as follows:

- The FOL will supervise and check daily that the field measurements are made accurately, equipment is thoroughly decontaminated, samples are collected and handled properly, and fieldwork is documented accurately and neatly.

- The TOM will maintain contact with the FOL and Data Validation Coordinator to ensure that management of the acquired data proceeds in an organized and expeditious manner.

Details regarding audit responsibilities, frequency, and procedures are discussed in the remainder of this section. Field performance and system audits are discussed in Section 10.1. Laboratory performance and system audits are discussed in Section 10.2.

10.1 FIELD PERFORMANCE AND SYSTEM AUDITS

This section discusses internal and external field performance and system audits.

10.1.1 Internal Field/Office Audits

10.1.1.1 Internal Field/Office Audit Responsibilities

In addition to the daily checks performed by the FOL, an independent performance and system audit of field activities may be conducted by the TtNUS Quality Assurance Manager (QAM) or designee. Such audits are scheduled as part of the NSWC Crane RCRA environmental investigation program, which includes this and other environmental projects, with individual projects being selected for audit by the QAM without the involvement of TOM. If a formal field audit is conducted for this study, the QAM (or designee) will be responsible for ensuring that sample collection, handling, and shipping protocols, as well as equipment decontamination and field documentation procedures, are being performed in accordance with the approved project plans and SOPs.

10.1.1.2 Internal Field/Office Audit Frequency

An internal field audit will be conducted at the beginning of the field program. This will allow for identification and correction of problems early in the program.

10.1.1.3 Internal Field Audit Procedures

The field audit will be conducted in accordance with the following procedures:

- Prior to the audit, the auditor will prepare a detailed checklist to be used as an auditing guide. An example audit checklist is provided in Appendix B of the QAPP.

- Upon arrival at the audit location, the auditor shall conduct a pre-audit meeting with the responsible management of the project to be reviewed.

- Field audits will include a review of required project documentation (logbooks, sample logsheets, etc.) and field operations (sample COC, sample handling, etc.) to evaluate completeness and compliance with applicable SOPs.

- The audit checklist will be used to record observations including any noted nonconformances.

- A formal post-audit debriefing will be conducted, and potential immediate corrective actions will be discussed.

- The auditor will generate an audit report that addresses corrective actions. This report will be provided by the auditor to the TOM.

- The TOM will ensure that all corrective actions are addressed and will provide written verification of corrective action implementation to the auditor.

- The auditor will manage corrective action verification and audit closure.

- The following audit records will be maintained by the QAM:

  - Audit checklists
  - Audit reports

- Response evaluations
- Verification of corrective actions
- Follow-up checklists and audit reports

The results of the audit will be considered acceptable if all SOPs and project planning document requirements are followed. If problems are identified, corrective action will be initiated in accordance with the procedures outlined in Section 13.0.

10.1.2 External Field Audits

External field audits may be conducted by the Indiana Department of Environmental Management (IDEM), U.S. EPA Region 5, or both. Details regarding field audit responsibilities, frequency, and procedures are at the discretion of the agencies.

10.1.2.1 External Field Audit Responsibilities

External field audits and associated responsibilities are at the discretion of IDEM or U.S. EPA Region 5.

10.1.2.2 External Field Audit Frequency

External field audit frequency is at the discretion of IDEM or U.S. EPA Region 5.

10.1.2.3 Overview of External Field Audit Process

The external field audit process is at the discretion of IDEM or U.S. EPA Region 5.

10.2 LABORATORY PERFORMANCE AND SYSTEMS AUDITS

10.2.1 Internal Laboratory Audits

Internal laboratory audit responsibilities, frequencies, and procedures are discussed in this section.

10.2.1.1 Internal Laboratory Audit Responsibilities

The subcontract laboratory QA/QC Officer (or designee) performs routine internal audits of the laboratory. Internal laboratory audits are also conducted by the U.S. Navy. TtNUS holds no responsibility for such audits. Performance and system audits of laboratories are coordinated through the NFESC by an independent QA contractor. The NFESC and its contractor are responsible for ensuring that the

subcontracted laboratories comply with good laboratory practices and the general requirements of all analytical services provided by the laboratory.

10.2.1.2 Internal Laboratory Audit Frequency

At a minimum, the subcontract laboratory conducts internal system audits of each laboratory analytical department on an annual basis. Internal audits are performed bi-annually at Laucks Testing Laboratories, Inc., if no external audits are conducted. In addition, each laboratory department at Laucks Testing Laboratories, Inc., analyzes blind performance evaluation (PE) samples as described in SOP LTL-1009 (Appendix A). Data audits are also performed by the Laucks Testing Laboratory QA/QC Officer at a minimum frequency of once per year for each analytical area.

Internal laboratory performance and system audits are completed by the U.S. Navy for each contracted laboratory in the NFESC QA Program on an 18-month schedule.

10.2.1.3 Internal Laboratory Audit Procedures

Internal systems audits are conducted to detect any problems in sample flow, analytical procedures, or documentation and to ensure adherence to laboratory SOPs. Laucks Testing Laboratories, Inc., internal audit procedures are described in SOP LTL-1017 (Appendix A).

Internal U.S. Navy laboratory audit procedures, as performed by a Navy contractor, include a pre-screening process that requires review of the laboratory's QA manual, analysis of PE samples, generation of data deliverables for the PE samples, an on-site technical systems audit of the laboratory, and satisfactory resolution of all deficiencies and findings.

10.2.2 External Laboratory Audits

This section discusses external laboratory audit responsibilities, frequencies, and procedures.

10.2.2.1 External Laboratory Audit Responsibilities

External laboratory audits may be performed at the discretion of IDEM or U.S. EPA Region 5. Each laboratory is also involved in external audits and PE studies throughout the year, as required, to maintain certifications or approvals by other regulatory agencies or programs. U.S. EPA Region 5 has recently audited Laucks Testing Laboratories, Inc. for another related U.S. Navy Crane project and the outcome was favorable.

10.2.2.2 External Laboratory Audit Frequency

An external laboratory audit may be conducted by IDEM or U.S. EPA Region 5 prior to or during sampling and analysis activities.

10.2.2.3 External Audit Procedures

External audit procedures are at the discretion of IDEM or U.S. EPA Region 5. External laboratory audits may include (but are not limited to) review of laboratory analytical procedures, laboratory on-site audits, and submission of PE samples to the laboratory for analysis.

11.0 PREVENTIVE MAINTENANCE PROCEDURES

Measuring equipment used in environmental monitoring or analysis for the NSWC Crane SWMU 30 RFI shall be maintained in accordance with the manufacturer's operation and maintenance manuals. Equipment and instruments shall be calibrated in accordance with the procedures, and at the frequency discussed in Section 6.0 (Calibration Procedures and Frequency). Preventive maintenance for field and laboratory equipment is discussed in the remainder of this section.

11.1 FIELD EQUIPMENT PREVENTIVE MAINTENANCE

Preventive maintenance of field equipment is described in Section 2.4 of the Work Plan. The TtNUS Equipment Manager and the instrument operator will be responsible for ensuring that equipment is operating properly prior to use and that routine maintenance is performed and documented. Any problems encountered while operating the instrument will be recorded in the field logbook, including a description of the symptoms and corrective actions taken. If problem equipment is detected or requires service, the equipment should be logged, tagged, and segregated from equipment in proper working order. Use of the instrument will not be permitted until the problem is resolved.

11.2 LABORATORY INSTRUMENT PREVENTIVE MAINTENANCE

Proper maintenance of laboratory instruments and equipment is essential to ensuring their readiness when needed. Dependent on manufacturer's recommendations, maintenance intervals are established for each instrument. All instruments must be labeled with a model number and serial number, and a maintenance logbook must be maintained for each instrument. Personnel must be alert to the maintenance status of the equipment they are using at all times. Table 11-1 summarizes preventive maintenance procedures performed by Laucks Testing Laboratories, Inc., for key analytical instruments and equipment.

The use of manufacturer-recommended grades (or better) of supporting supplies and reagents is also a form of preventive maintenance. For example, gases used in the various instruments must be of sufficient grade to minimize fouling of the instrument. The routine use of septa, chromatographic columns, and other supporting supplies from reputable manufacturers will assist in averting unnecessary periods of instrument downtime. An inventory of critical spare parts will also be maintained by the laboratory to minimize instrument downtime.

TABLE 11-1

**PREVENTIVE MAINTENANCE FOR ANALYTICAL INSTRUMENTS
NAVAL SURFACE WARFARE CENTER
CRANE, INDIANA**

Instrument	Preventive Maintenance	Maintenance Frequency
GC/MS - Volatiles	Change pump oil.	Yearly.
	Clean and rinse transfer lines, trim front end of column, rinse 6-port valve, clean or replace sample lines, replace trap, replace column, clean source, replace fittings, change sample block on autosampler, replace filaments.	As needed.
HPLC	Change filter frit in mixer.	As needed (when pressure builds).
	Change column pre-filter.	As needed (2 to 3 months).
	Rinse water pump with methanol, filter water, sonicate water intake filter frit.	Approximately weekly.
	Change pump seals.	As needed.
ICP/MS	Clean or change air filters, change pump oil.	Semi-annually.
	Clean torch, replace nebulizer tips, replace pump tubing, replace injector, change cones.	As needed.
	Check mass calibration.	Every 2 weeks.
	Check sensitivity.	Daily.
Mercury Analyzer	Check and replace pump tubing, check and replace membrane, check and clean windows.	As needed.
Spectro-photometer	Clean sample compartment and entrance windows.	Semi-annually.
	Check wavelength calibration.	Annually.
Lachat Ion Analyzer	Lubricate pump.	Semi-annually.
	Replace pump tubing.	As needed (~ 1 to 2 months)
	Change cadmium column.	As needed.
Refrigerators	Monitor temperature.	Daily.

12.0 SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA PRECISION, ACCURACY, AND COMPLETENESS

Compliance with the quantitative QC objectives for laboratory accuracy and precision, as outlined in Tables 3-1 through 3-8 of Section 3.0, will be evaluated during data validation. Compliance with the field data precision specification in Section 3.1.2 will also be evaluated for field duplicates during data validation. The validation process will be used to flag with quality indicators data that fall outside the QC acceptance limits. Compliance with the completeness objectives for field and laboratory data will be completed manually (field data) and electronically via a database subroutine (laboratory data). Sections 12.1, 12.2, and 12.3 present equations to be used for computing precision, accuracy, and completeness values, respectively.

In general, data validation requires that data be evaluated batch-by-batch based on the results of quality indicators for the respective batches. Section 12.4 presents additional data quality considerations that will be evaluated after completion of data validation. These additional considerations are designed to incorporate data quality factors that extend evaluation of the simple quantitative estimators for precision, accuracy, and completeness.

12.1 ACCURACY ASSESSMENT

As discussed in Section 8.2, a minimum of 1 of every 20 samples is spiked with a known amount of the analyte or analytes to be evaluated to assure the accuracy of the analytical procedures. The spiked sample is then analyzed. The increase in the analyte concentration observed in the spiked sample, due to the addition of a known quantity of the analyte, compared to the reported value of the same analyte in the unspiked sample determines the %R. Control charts are plotted for each commonly analyzed compound and kept on matrix-specific and analyte-specific bases. The %R for a spiked sample is calculated by using the following formula:

$$\%R = \frac{\text{Amount in Spiked Sample} - \text{Amount in Sample}}{\text{Known Amount Added}} \times 100\,\%$$

Section 8.2 also discusses the use of surrogate spikes and LCSs as measures of accuracy. The %R for a surrogate spike or LCS is calculated by using the following formula:

$$\%R = \frac{\text{Experimental Concentration}}{\text{Known Amount Added}} \times 100\,\%$$

12.2 PRECISION ASSESSMENT

As discussed in Section 8.2, laboratory duplicate samples (for inorganic analyses) and MSD samples (for organic analyses) are prepared and analyzed at a minimum frequency of 1 per every 20 environmental samples. As discussed in Section 3.6, field duplicate samples will also be collected at a minimum frequency of 1 per 10 environmental samples. The RPD between the sample (or spike) and duplicate (or duplicate spike) is calculated by using the following formula:

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

12.3 COMPLETENESS ASSESSMENT

As discussed in Section 3.3, completeness is the ratio of the number of valid sample results to the total number of samples planned. Following the completion of the analytical testing and data validation, percent completeness will be calculated by using the following equation:

$$\text{Completeness} = \frac{\text{(number of valid measurements)}}{\text{(number of measurements planned)}} \text{ X } 100\,\%$$

The results of the data validation process and the completeness assessment will be summarized in the SWMU 30 Ground Water Assessment Report.

12.4 DATA ASSESSMENT

The field and laboratory data collected during ground water monitoring for SWMU 30 will be used to meet the objectives presented in Table 1-1. The data obtained during each round of sampling and analysis will be both qualitatively and quantitatively assessed on a parameter-specific basis. The results of these assessments will be presented in quarterly reports and a final Ground Water Assessment Report. Examples of issues to be considered in the data assessments are as follows:

- Were all samples obtained using the methodologies specified in the Work Plan?
- Were samples obtained from all sampling locations for all analyses specified in the Work Plan?

- Were all field analyses performed using the methodologies specified in the Work Plan?
- Were all laboratory analyses performed using the methodologies and SOPs specified in the QAPP?
- Were all data validated as specified in the QAPP?
- Were any data points determined to be unusable (qualified as "R") during the data validation process? If so, are these rejected data points critical in meeting the DQOs for the project?
- Do any analytical results exhibit elevated quantitation limits? If so, what are the causes and what overall impact does this have on the data?
- Did coelution of any chromatographic peaks occur during analysis for explosives? If so, what overall impact does this have on the data?
- Have sufficient data of appropriate quality been generated to support statistical analysis?

13.0 CORRECTIVE ACTION

The TtNUS QA/QC program requires that any and all personnel noting conditions adverse to quality report these conditions immediately to the TOM and QAM. These parties, in turn, are charged with performing root-cause analyses and implementing appropriate corrective action in a timely manner. The QAM is ultimately responsible for documenting all findings and corrective actions taken and for monitoring the effectiveness of the corrective measures performed.

13.1 FIELD CORRECTIVE ACTION

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

Corrective action may include resampling and may involve amending previously approved field procedures. Minor modifications to field activities, such as the collection of additional samples, will be initiated at the discretion of the FOL, subject to on-site approval by the NSWC Crane Environmental Protection Department, and documented in the field log book. Approval for major modifications (e.g., elimination of a sampling point) must be obtained and documented via a Field Task Modification Request (FTMR); Navy (in conjunction with U.S. EPA Region 5 and IDEM) approvals will be obtained. The FOL is responsible for initiating FTMRs. An FTMR will be initiated for all deviations from the project plan documents, as applicable. An example of an FTMR is provided as Figure 13-1. Copies of all FTMRs will be maintained with the on-site project planning documents and will be placed in the final evidence file.

13.2 LABORATORY CORRECTIVE ACTION

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

FIGURE 13-1

Tetra Tech NUS, Inc.
FIELD TASK MODIFICATION REQUEST FORM

Client Identification Project Number TMR Number

To_____Location_____Date_____

Description:

Reason for Change:

Recommended Disposition:

Field Operations Leader (Signature, if applicable) Date

Disposition:

Task Order Manager (Signature, if required) Date

Distribution:
Program Manager
Quality Assurance Officer
Task Order Manager
Field Operations Leader

Others as required_____

- QC data are outside established warning or control limits;
- method blank analyses yield concentrations of target analytes above acceptable levels;
- undesirable trends are detected in spike recoveries 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 audits or from performance evaluation sample test results.

Any corrective action taken above the analyst level that cannot be performed immediately at the instrument will be documented. Corrective actions are typically documented for out-of-control situations on a corrective action form or an out-of-control event form. Copies of the forms used by Laucks Testing Laboratories, Inc., are included as Appendices 1 and 3 of SOP LTR-1008, which is in Appendix A of this QAPP. Further details describing the system used by Laucks Testing Laboratories, Inc., to identify, document, and resolve out-of-control events are provided in SOP LTR-1008.

13.3 CORRECTIVE ACTION DURING DATA VALIDATION AND DATA ASSESSMENT

The need for corrective action may become apparent during data validation, interpretation, or presentation activities, or problems may be identified as a result of oversight findings. The performance of rework/resampling, instituting 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 corrective action.

14.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

QA reports to management will be provided in various formats during the course of the NSWC Crane SWMU 30 RFI. Data validation letter reports will be prepared on a SDG-specific basis and will summarize QA issues for the subcontract laboratory data. During the field investigation, the FOL will make daily reports to the TOM via telephone summarizing accomplishments and QA/QC issues. Monthly progress reports will be prepared by the TOM. At the request of the Navy RPM, these reports may be distributed to the Base's RPM or the appropriate regulatory agency. QA reports will also be prepared by the subcontract analytical laboratory as QC limits are updated or if significant plan deviations result from unanticipated circumstances.

14.1 CONTENTS OF PROJECT QUALITY ASSURANCE REPORTS

The data validation reports 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., holding time exceedances or calibration noncompliances) the Data Validation Coordinator will notify the TOM, QAM, Technical Program Manager, Deputy Program Manager, and Laboratory Services Coordinator. Such notifications (if necessary) are typically provided via internal memoranda and are placed in the project file. Such reports contain a summary of the noncompliance, a synopsis of the impact on individual projects, and recommendations regarding corrective action and adjustments. Corrective actions are initiated at the program level.

The FOL will provide the TOM with daily reports via telephone during the course of each sampling event. These reports will discuss accomplishments, deviations from the Work Plan, upcoming activities, and QA. The TOM 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 subcontract analytical laboratory will provide QA reports to TtNUS whenever QC limits for parameters associated with the NSWC Crane SWMU 30 RFI are updated or if significant plan deviations result from unanticipated circumstances. Because MDLs, IDLs, and RLs, as applicable, will be included in the analytical data packages for NSWC Crane samples, it is not necessary for the laboratories to include updated MDLs, IDLs, and RLs in the QA reports unless the updates result in RLs that exceed risk-based target levels.

14.2 FREQUENCY OF QUALITY ASSURANCE REPORTS

The following frequencies will apply to QA reports for the NSWC Crane SWMU 30 RFI: (1) data validation QA reports - contingent on SDG delivery data; (2) field progress reports - daily during the course of the field investigation; (3) monthly progress reports – monthly; and (4) laboratory QA reports – as required based on QC limit updates or the occurrence of plan deviations resulting from unanticipated circumstances.

14.3 INDIVIDUALS RECEIVING/REVIEWING QUALITY ASSURANCE REPORTS

Data validation QA reports will be provided to the TOM for inclusion in the project files. In the event that major problems are observed for a given laboratory, the Technical Program Manager, Deputy Program Manager, QAM, TOM, and Laboratory Services Coordinator will be provided with copies of the QA report. Daily field progress reports via telephone will be provided to the TOM. Monthly progress reports will be provided to the U.S. Navy. Laboratory QA reports will be provided to the TOM; these reports will be forwarded to U.S. EPA Region 5 only if QC updates result in RLs that exceed risk-based target levels, if QC limits for key parameters (e.g., metals or other analytes associated with the site based on site history) degrade significantly, or if any significant plan deviations result from unanticipated circumstances. Copies of any cited QA reports will be provided to U.S. EPA Region 5 immediately upon request.

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U.S. EPA Region 5, April 1998. Region 5 QAPP Policy. Waste, Pesticides, and Toxics Division, Chicago, IL.

APPENDIX A

STANDARD OPERATING PROCEDURES

APPENDIX A

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SOP Number	Revision	Title
Laucks Testing Laboratories, Inc. SOPs:		
• LTL-1001	3	Elements of SOP and Method Formats
• LTL-1002	3	Document Tracking and Control
• LTL-1003	2	Chain-of-Custody and Documentation Procedures
• LTL-1004	3	Documentation of Analyst Competence and Training
• LTL-1005	4	Analytical Balances
• LTL-1006	2	Refrigerator, Freezer, and Oven Thermometer Calibration and Maintenance
• LTL-1007	2	Use of Instrument Records and Logbooks
• LTL-1008	5	QC Corrective Action
• LTL-1009	2	Blind Spike Program
• LTL-1011	4	Procedures for the Determination and Reporting of Detection Limits, Reporting Limits, Precision and Accuracy Studies, and Control Limits.
• LTL-1012	1	Solvent QC Monitoring for Trace Residue Analysis
• LTL-1013	3	Preparation, Storage, Shelf Life and Traceability Documentation of Standards and Reference Materials
• LTL-1017	1	Internal Audit Procedures
• LTL-1018	0	Overview of Review and Approval Practices for Validatable Data Packages
• LTL-1019	1	Controlling, Maintaining, and Monitoring Laboratory Logbooks
• LTL-1020	1	Integration of IC, GC, HPLC and GCMS Peaks
• LTL-2001	5	Waste Segregation and Disposal
• LTL-3077	3	Extraction Method for Ordnance Compounds in Water (8330)
• LTL-4002	5	Electronic Sample Entry and Log-In
• LTL-4103	0	Reviewing a Sample Delivery Group (SDG) and Updating Project in the LIMS
• LTL-4201	0	Package Deliverables for all Reporting Levels
• LTL-7003	2	Inorganic Glass Cleaning Procedure
• LTL-7009	2	Microwave Assisted Acid Digestion of Aqueous Samples and Extracts Using SW846 Method 3015
• LTL-7010	2	Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FLAA or ICP Spectroscopy by SW846 Method 3010A
• LTL-7202	3	Metals Analysis Using Inductively Coupled Plasma - Mass Spectrometry (ICP/MS) SW846 Method 6020
• LTL-7501	1	Operation of the PE FIMS 400 for the Analysis of Hg
• LTL-8000	1	Determination of Retention Time Windows
• LTL-8265	1	Determination of Volatile Organic Compounds by Method SW 8260B
• LTL-8302	1	HPLC Ordnance 8330 Data Review
• LTL-8330	9	Determination of Nitroaromatics and Nitramines by SW-846 Method 8330
• LTL-9104	2	Preparation and Analysis of Total and Amenable Cyanide according to EPA Methods 335.1, 335.3, and SW 846 Method 9012A

APPENDIX A

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SOP Number	Revision	Title
Laucks Testing Laboratories, Inc. SOPs:		
• LTL-9108	1	Phosphorus Analysis, All Forms
• LTL-9109	1	Ammonia Analysis by Lachat Auto-Analyzer, EPA Method 350.1
• LTL-9125	1	EPA 353.2 Nitrate/Nitrate as N by Lachat
• LTL-9133	0	Determination of Total Kjeldahl Nitrogen in Water by EPA 351.2
TtNUS (formerly B&R Environmental) SOPs:		
• CT-05	0	Database Records and Quality Assurance

LAUCKS TESTING LABORATORIES, INC.

SOPs

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP No. LTL-1001

Previous SOP No. LTL-0001

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

Rev. 3

Laucks is in the process of re-numbering our SOPs. As an interim measure, this page serves as the cover page for those SOPs whose header information has not been updated. This page details the title, the SOP number that it is being controlled under, and the previous SOP number. The previous SOP cover sheet has been manually corrected to reflect the change but each page header will reflect the old numbering system. As SOPs are revised, the full header and cover page will be updated.

UNCONTROLLED

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #~~LTL-0001~~ 12/24/95
LTL-1001

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

Revision history:

Number	Date
3.0	7/22/94
2.0	9/16/87
1.0	7/31/86

Written by: Harry Romberg, QC Officer Date: 7-25-94

Reviewed by: Mike Nelson, Technical Director Date: 7/25/94

Approved by: John Buerger, Laboratory Director Date: 7/25/94

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1. Introduction and Scope

1.1. The purpose of this SOP is to define the process of creating SOPs and Methods. SOPs are considered 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. Both have similarities and differences in structure and necessary elements. 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.

2. Procedures

2.1. Word Processing Formats

2.1.1. Two word processing formats are currently allowed, Word for Windows 2.0 or higher being the preferred format for authors with access to a computer with this capability, and XyWrite for authors who either do not have Word for Windows capability or who are completing an SOP already in XyWrite at the time of this writing. **NO other word processing formats are allowed.**

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-0054. This form can be obtained from the QC Department.

2.2.2. SOPs are considered 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. At the time of initiation of a new document, an SOP or Method number will be assigned by the QC Officer or designee. SOPs will be prefixed with the letters LTL- (such as LTL-0001). Methods will be prefixed with the letters LX- (such as LX-0123).

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 1.0 with subsequent revisions generally being incremented by 1 (2.0, 3.0, etc.)

2.2.4. The author should then obtain a copy of the template to be used for writing the SOP or Method. Hardcopies of these formats are located in Appendices to this SOP. These formats may change somewhat without updating of this SOP so the author should obtain the latest revision

from the QC Department in order to ensure that the latest version is being used. 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. Electronic copies may be obtained in several ways.

2.2.5. If a Word document is being created, the template may be accessed from the projects drive (P: drive on most people's computer systems) in the SOP directory.

2.2.6. It may also be obtained directly from the QC Department. The Word versions are identified as .DOC files and the XyWrite versions by the .XY extension.

2.2.7. On some computers, the template is stored as a template file. It may be easily accessed by choosing [File][New] and the template name (SOPhead, Iorgtemp or Orgtemp). All of these templates are written in Word. These templates were written primarily as guidelines for comprehensive writing of an SOP or method. 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.

2.2.8. SOPhead is the very 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. Specific elements of a method are outlined below and in the method templates Iorgtemp and Orgtemp.

2.2.9. 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.

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

2.2.11. If a previous SOP or Method in any format is being revised, it MUST be obtained from the QC Department in order that the most recent previous version is being used for the revision.

2.2.12. If a XyWrite document is being written or revised, the template must be obtained from the QC Department. Where at all possible, the Word format is preferred. Unless only minor alterations are being made in a revision, or the author does not have access to a Windows based computer with Word for Windows, as specified above, it is likely that the revision will be required to be made in the Word format.

2.2.13. The template should be opened and appropriate information filled in. If the Word version of a Method is being used, most of the items which should need input are highlighted in red. 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.

2.2.14. A Word document may be opened by selecting the [File][Open] selection and completing the requested information. By selecting the proper drive and then the appropriate directory, the template (and possibly some other documents) should appear in the filename box. Select the template file and select [OK].

2.2.15. A XyWrite document may be opened by calling it from the command line by typing [ca {path}{filename}] and [Return] or [F9]. Alternatively, the path can be selected by typing [cd {path}] and [Return] at the command line, just like a DOS prompt. The author can then see the file by doing a directory [dir] and then call it, [ca {filename}].

3. Elements Of An SOP/Method

3.1. Elements

3.1.1. SOP formats are more general and free-form, not requiring the same specific elements as a Method. SOPs need only have the appropriate cover and header information, signature sheet, 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.

3.1.2. Methods contain the appropriate cover and header information, signature sheet, 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. Required appendices include preparation of standard solutions, a Method QC Table and a procedural flow chart.

3.1.3. 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 QC Officer)
The title of the SOP including EPA method number reference when appropriate
The revision history (revision number and date of approved revision)
Signature of Author and date signed

Signature of managerial reviewers (Divisional Manager, Technical Director, QC Officer, Lab Director)

3.1.4. All of the SOP/Method templates contain an Operators Statement. The operators statement must include the header information present on the body of the SOP/Method. In addition, it must also contain the title of the document and columns for the operators signatures, printed name, and date signed. This page is to be signed by all persons who will be undertaking the operation for which the document was written. The operator(s) are expected to read and understand the process described before it is undertaken.

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

3.1.6. All of the SOP/Method templates contain a header record which identifies the SOP/Method number, revision, date, and the method or revision it replaces (if any). This record is not readily apparent in the "normal" mode of either Word or XyWrite templates but must be completed by the author.

3.1.7. In Word, choose [View][Header/Footer][Header] and [OK]. 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 changing the appropriate selection.

3.1.8. In XyWrite, one must find the triangle which, when the cursor is placed on it, appears as [RHA etc.] on the line immediately below the command line. Place the cursor on that triangle and select [control F3]. When through modifying the header, press [F3] to close the triangle. **Do not** delete this triangle and attempt to re-create the header or it will likely not meet the SOP/Method header criteria.

3.1.9. All SOPs/Methods contain an Introduction and Scope. The following sections should be contained in this section:

3.1.10. 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.

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

3.1.12. A part defining terms, particularly those which are specific to that procedure and may not be familiar to all readers.

3.1.13. 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.

3.1.14. 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 (LTL-0032) for final disposal.

3.1.15. 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. 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.

3.1.16. 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.

3.1.17. 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.

3.1.18. 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?")

3.2. Saving the Document

3.2.1. Save the document under another name than it was called when it was opened. In most instances, the template will be a read-only file and trying to overwrite it will produce an error message.

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

3.2.3. In XyWrite, this is accomplished by typing [sa {filename}] or [st {filename}] on the command line ([F5] or [F6] gets you to the command line) and [Enter] or [F9] to execute the command. Note: in XyWrite, [st] saves and closes the file (clears the screen), whereas [sa] saves the file but keeps it open for possible further editing.

3.2.4. Note that in either word-processing format, 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.

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 passed to the QC Officer for review and distribution. An unapproved SOP or Method document is not considered official.

Appendix I

SOP Template (WORD)

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-0000

Title: (Place title here)

Revision history:

Number	Date

Written by: _____ Date: _____
(Author's name typed here & signed above)

Reviewed by: _____ Date: _____
(Supervisor's name & title, delete if not applicable)

Reviewed by: _____ Date: _____
Mike Nelson, Technical Director

Reviewed by: _____ Date: _____
Harry Romberg, QC Officer

Approved by: _____ Date: _____
John Buerger, Laboratory Director

Title: (Place title here)

Operator's signature page

Signature	Printed Name	Date

Signature page, for department manager's files

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    1.2. Definition of Terms 4
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1. Introduction and Scope

1.1. Method Description

1.1.1. Describe the general process, limitations, applicability, and any other general topics. No need to get specific here except to describe where this process may vary from a published procedure.

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 analysis.

1.2. Definition of Terms

1.2.1. This section defines terms and acronyms as they are used in this SOP. This section or parts of it may be deleted if the analysis is basic and has no terminology that may be confusing to anyone likely to read the procedure.

2. Equipment List and Standards

2.1. Equipment

2.1.1. Describe the Instrument or instrumental system, data system, and other relevant equipment. This section may or may not be applicable. Delete it if not appropriate.

2.2. Reagents

2.3. Standards

3. Safety precautions and Waste Disposal

3.1. Safety Precautions

3.1.1. Only keep the applicable parts of this section (if any). Add others that may be applicable.

3.1.2. All standards, samples and sample solutions should be handled as if they are hazardous substances.

3.1.3. Refer to the instrument manufacturer's manual for routine instrument precautions.

3.1.4. Routine precautions include an awareness of the moving parts on the instrument you're using. These parts are often charged with power from an electrical component or with high pressure gas and have the potential to do harm if not used properly.

3.1.5. Electrical shock - All instruments present the possibility of electrical shock The operator should take all precautions including ensuring that all instruments are operated with fully grounded power outlets, turning off the instrument and disconnecting the instrument from the electrical power supply before working on any electrical components, etc.

3.1.6. Anything that might be hazardous (working with fire, acid, toxics, etc.) should be listed here and at the point in the procedure where the hazard might exist. Precautions which you feel are particularly appropriate should be elaborated upon in this section.

3.2. Waste Disposal

3.2.1. Describe all waste disposal practices associated with this analysis. It is not necessary to describe what happens to waste after it has been collected if that waste is being sent out for recycling or disposal. This is covered in our waste disposal SOP.

3.2.2. Waste segregation and disposal from the point of collection is further covered in Laucks SOP LTL-0032.

4. Calibration and Quality Control

4.1. Describe any specific quality control procedures, frequency, criteria, and corrective actions here. One would expect that many procedures will have elements of quality control. Although this section may be deleted if inappropriate, the author should consider what quality control procedures are or should be involved with whatever process is being described.

5. Operation procedures

5.1. Describe the actual process here. Where the introduction and scope was general, this section should be very specific so that any person reasonably familiar with what is going on can get specific instructions here and complete the task with reasonable assurance that it will be done properly.

6. Reports

6.1. Data Package Organization

6.1.1. It may be more appropriate to title this section differently depending upon the process being described. Any basic paperwork and/or data associated with this process should be described here including both the organization and the contents. This is

Appendix I

Flow Chart

Appendix II

(Title here)

Add any other appendices that may be applicable to your method.

Appendix II (WORD)

Inorganics Method Template

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LX-0000

Title: (Place title here)

Revision history:
Number Date

Written by: _____ Date:_____
(Author's name typed here & signed above)

Reviewed by: _____ Date:_____
(Supervisor's name & title, delete if not applicable)

Reviewed by: _____ Date:_____
Mike Nelson, Technical Director

Reviewed by: _____ Date:_____
Harry Romberg, QC Officer

Approved by: _____ Date:_____
John Buerger, Laboratory Director

Title: (Place title here)

Operator's signature page

Signature	Printed Name	Date

Signature page, for department manager's files

1. Introduction and Scope

1.1 Method Description

1.1.1 Describe the method, limitations of the method, applicable matrices, analytes and their detection limits.

This method is restricted to use by, or under the supervision of analysts experienced in the technique described. Each analyst performing this method must have demonstrated the ability to perform the described analysis.

1.2 Sample Collection, Sample Storage, Holding Times

Describe containers, preservation, storage conditions (refrigerated at 4 C, etc.), and holding times to preparation and/or analysis based on the date of collection. Only EPA CLP calls for holding times calculated from the Verified Time of Sample Receipt (VTSR). In-house CLP or any analyses other than for the EPA CLP program are calculated from date of **collection**.

1.3 Definition of Terms

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. This section or parts of it may be deleted if the analysis is basic and has no terminology that may be confusing to anyone likely to read the procedure.

Blank spike - A background free matrix (DIW for water, clean sand for soils/sediments) to which known amounts of target analytes are added each time samples are prepared. Blank spikes are required on all HAZWRAP and NEESA work. Note that an LCS or SRM (see below) will substitute as a blank spike for most inorganic analyses. 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 (almost always after every 10 samples) during the analysis sequence to determine whether the instrument or system has remained in calibration.

CLP - Contract Laboratory Program - The USEPA program that contracts with laboratories to provide laboratory services. The term has come to mean a much broader set of

methods and deliverables. In context of this SOP, CLP means procedures or operations which are detailed in the CLP contract and which are extended to a broader working definition.

Corr Coef, CC - Correlation coefficient - A measure of the "goodness of fit" of a set of data to a regression model. The closer the value is to 1, the higher the degree of confidence in the correlation

DIW - Deionized water - Lab reagent water. This water should be free of virtually all analytes.

ICB - Initial calibration blank - This term is borrowed from CLP. An instrument blank is made up in the same way as calibration standards, without target analytes.

ICV - Initial calibration verification - This term is borrowed from the CLP protocol. It is a standard which is analyzed at the start of each analytical run that is compared to the initial multi-point calibration to determine whether the instrument calibration is accurate.

IDL - Instrument detection limit. The lowest concentration of a target analyte that will yield a signal:noise ratio of least 3x. Used as a starting point for selecting MDL study spiking levels.

MDL - Method detection limit - The lowest concentration a sample which will yield a positive result that is greater zero at a known level of confidence. MDLs are empirically determined by Laucks.

MDL standard - Method detection limit standard - A standard prepared so that the concentrations of the target analytes are no greater than 4x the empirically determined MDLs. This standard is used to verify that the instrument or system is capable of detecting the target analytes on an ongoing basis.

QC check standard - Quality control check standard. Referred to in this SOP as a blank spike. A QC check standard is used to determine whether the analytical system is in control if MS/MSD recoveries are out of control. See also blank spike.

SRM or LCS - Standard Reference Material or Laboratory Control Sample. This is a material of approximately the same matrix as the samples, containing a known and usually certified amount of target analyte and which is prepared and analyzed in the same manner as a typical sample. This sample is used to demonstrate that the analytical system is in control. It may be considered to be a blank spike for most inorganic analyses and is preferred over artificially spiking blank materials.

QC period - Quality control period - An analysis sequence initiated by the analysis of one or more standards, followed by samples, and terminated with a standard and blank analysis. A QC period can be open-ended chronologically, but calibration verification must be documented using the procedures in this SOP

RSD or %RSD - Relative standard deviation or percent relative standard deviation - The ratio of the standard deviation of a set of values to the mean of the set of values. A measure of the similarity of the values one to another.

RT - Retention time - The time (in minutes) at which a target analyte elutes from the IC (or other) column.

RT window - Retention time window - The +/- value which is applied to the ICV to establish the time range used to make tentative target analyte identifications.

Sequence - A set of sample extracts and standard solutions injected into an instrument in a chronologically continuous group. See also QC period.

Equipment List and Standards

Instrument

Describe the instrument or instrumental system, data system, and other relevant equipment.

Standards

Describe the exact preparation of all required standards. Tabulate the concentrations of all analytes in all standards prepared. Include all stock, intermediate, and working solutions for calibration mixes, blank spike mixes and MS/MSD mixes. Be sure to describe the MDL standard and the ICV/CCV solutions.

In Appendix 1, detail all solutions. List all target analytes and the concentrations desired.

Safety precautions and Waste Disposal

Safety Precautions

All standards, samples and sample solutions should be handled as if they are hazardous substances.

Refer to the instrument manufacturer's manual for routine instrument precautions.

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.

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.

Anything that might be hazardous (working with fire, acid, toxics, etc.) should be listed here and at the point in the procedure where the hazard might exist. Precautions which you feel are particularly appropriate should be elaborated upon in this section.

Waste Disposal

Describe all waste disposal practices associated with this analysis. It is not necessary to describe what happens to waste after it has been collected if that waste is being sent out for recycling or disposal. This is covered in our waste disposal SOP.

Waste segregation and disposal from the point of collection is further covered in Laucks SOP LTL-0032.

Calibration and Quality Control

Method Detection Limit Study

Prior to the analysis of any samples, it is necessary to establish method detection limits. This procedure is fully described in Laucks SOP LTL-0027. 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.

Initial Multi-Point Calibration

Analyze standard solutions using at least 5 different concentration levels. The calibration curve (concentrations, how to make it up, etc.) should be thoroughly described here. The lowest

concentration should be at a concentration near, but above, the method detection limit. The highest concentration should define the upper usable working range of the instrument. Analyze the standard solutions from the lowest concentration to the highest.

Criteria

Initial calibration data is evaluated using the correlation coefficient of a linear regression analysis. The correlation coefficient must be 0.995 or greater for a 5-point calibration. All CCVs and sample extract concentrations must be computed using the regression equation.

Corrective action

If the criteria are not met, the instrument must be recalibrated.

Initial Calibration Verification

Immediately after the calibration curve, analyze a standard from a source other than that from which the calibration material was obtained.

Criteria

The calculated concentration of the ICV must be within the limits supplied by the manufacturer or should not exceed 90%-110% of the true value if no limits are provided.

(The author of the SOP must correctly identify the appropriate limit. If the published method is explicit on this subject, we should be using the published criterion and not some "Laucks" derivative.)

Corrective action

If the ICV criteria are not met, no samples can be analyzed. Perform system maintenance and re-check the ICV. If the criteria still cannot be met, the system must be recalibrated.

Initial Calibration Blank

After the analysis of the ICV standard an instrument blank (ICB) is analyzed. The levels of target analytes in the CCB should not exceed twice the detection limit.

Corrective action

If the initial CCB contains target analyte levels above twice the detection limit, the system is out of control. The source of contamination must be identified and corrected before proceeding with the analysis.

Again, if the published method is specific, and the criteria can reasonably be met, it should not be deviated from.

Method Detection Limit Standard

After the analysis of the ICV and the ICB, but before the analysis of any sample extracts, an MDL standard is to be analyzed. The MDL standard is used to provide on-going verification of the ability of the system to detect analytes at a concentration near the method detection limit. The MDL standard should read between 67% and 220% of the true value. This limit may be overlooked if the recovery of this check standard is high. It must, however, be detected for the system to be considered in control.

Corrective Action

If target analytes are not detected, the analysis must be terminated until the problem has been solved. Alternatively, if the affected samples are well above the detection limit (ie bracketed by appropriate standards), they may be reported. No undetected values should be reported if the MDL standard for that analyte(s) is undetected.

Continuing Calibration Verification (CCV) and Blank (CCB)

A mid-range calibration standard is analyzed after every 10 samples. Immediately following the CCV, a blank solution is analyzed. In addition, this standard and blank must be the last samples analyzed in the run.

The CCV must fall within ± 10% of the true value.

The levels of target analytes in the CCB should not exceed twice the detection limit.

Corrective action

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.

If the initial CCB contains target analyte levels above twice the detection 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 CCBs which are within limits.

Method Blanks

Method blanks are used to verify contamination free reagents and apparatus. They are prepared with every set of samples prepared at the same time or at least one blank every 20 samples which ever is more frequent. Any analyte response above the detection limit is reported. Method blank control limits are that contamination should not exceed twice the detection limit (5x in limited instances)

Corrective action

Corrective action may necessitate re-preparation and re-analysis of the sample set. For example if an analyte were found in the blank but not in any of the associated samples then sample group may not require re-analysis. In addition, if sample levels exceed 10 times the blank, the level of contamination may be considered insignificant. In any case, if re-preparation and re-analysis is not being undertaken, the analyst must first discuss the issue with the Quality Control Officer. It is the laboratory's responsibility to ensure that method interferences caused by contaminants in acids, solvents, reagents, glassware, and other sample processing hardware leading to discrete artifacts and/or elevated baselines in the analytical run be minimized. In the extreme case of chronic contamination, blanks may have to be analyzed from each stage of the sample processing to determine the contamination source so it can be eliminated. In all cases where blank contamination exceeds the control limit, a narrative comment must be made which documents the corrective actions taken.

Method Blank Spikes

A method blank spike follows the same protocol as with the matrix spike analysis except that the spiking solution is added to a method blank solution instead of an actual sample. A method blank with added analytes is a method blank spike. A method blank spike is the same as a QC check standard. A blank spike OR a standard reference material (SRM) should be analyzed with most analysis types. The SRM is the preferred material and the blank spike should only be analyzed where an SRM does not exist or is not practical for routine use.

Corrective action

If the MS/MSD recoveries are out of control, the blank spike recoveries are examined to assess whether the method was in control during sample preparation and analysis. Re-prepare and reanalyze any samples for which both the matrix spike recoveries are low and out of control and for which the associated blank spike demonstrates out of control and low recoveries.

Matrix Spike

A sample is chosen at random from the samples to be analyzed, and an aliquot of spiking solution is added to this sample prior to preparation. The analyst should attempt to avoid selecting samples which are identified by the client as blanks. As the purpose of the matrix spike is to test the system under "typical" conditions, the analyst may also avoid selecting the most difficult sample of the batch for spiking. It is not always required that a matrix spike analysis be performed with each preparation/analysis batch, however, the minimum frequency for MS analysis is 1 each per 20 samples per matrix. This will be best accomplished by running one with every batch for many analyses. This matrix spike sample is used to evaluate the matrix effect of the sample upon recovery of the analytes. The recovery of spike analytes is calculated as follows:

$$\text{recovery} = \frac{(SS - S) * 100}{SS}$$

where:
SS = concentration in spiked sample
S = native concentration in unspiked sample

The recovery criteria are detailed in the current Control Limits Catalog and in the Quality Control Database (QC_DB) and will change from time to time.

Corrective action

Samples with spike recoveries outside control limits will be reviewed for possible corrective action. Corrective action will first involve recalculation, followed by possible re-preparation, and/or reanalysis. This process should also look at the recovery of at the recovery of matrix spiking compounds from the SRM and/or blank spike analysis. In all cases a narrative explanation of the condition is required to detail the corrective actions taken.

Matrix Spike Duplicate

The compound recovery criteria are identical to those for the matrix spike sample. In addition, the matrix spike duplicate is used measure method precision. This is done by computing the relative

percent difference (RPD) between the matrix spike and matrix spike duplicate recovery values. This calculation is as follows:

$$RPD = \frac{|S1 - S2| * 100}{(S1 + S2)/2}$$

where:
S1 = measured concentration for MS sample
S2 = measured concentration for MSD sample

RPD control limits are detailed in the current Control Limits Catalog and in the Quality Control Database (QC_DB) and will change from time to time.

Sample Duplicate

Criteria

Sample duplicates are required only when MS/MSDs are not practical (ie TSS & TDS), when the client requests, when CLP practices are employed, or when the method specifically calls for duplicates. At least one duplicate sample per 20 samples per matrix is required when matrix spikes are being performed. When duplicate analysis is all that is being performed or when requested (ie WTPH O&Gs) the frequency is 10%. RPD values are calculated in a manner similar to MS/MSD RPDs:□

$$RPD = \frac{|S1 - S2| * 100}{(S1 + S2)/2}$$

where:
S1 = measured concentration in the initial analysis
S2 = measured concentration in the duplicate analysis

The RPD control limits are detailed in the current Control Limits Catalog and in the Quality Control Database (QC_DB) and will change from time to time.

Corrective action

If a trend in out of control RPD values is observed, the methods used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable reproducibility. 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.

Operation procedures

Sample Analysis

Analysis sequence

A detailed analysis sequence should be provided here or added as an appendix.

Instrumental Conditions

Describe the exact conditions for the analysis including all temperatures, flows wavelengths, pump rates, etc.

Analytical Operation

Actual operation procedures should be discussed in this section (how to process samples, standards, etc. Standards curves, calibration, etc. are discussed in a following section. The actual procedure (add reagent A to sample, mix and wait 10 minutes....etc.) should be detailed here.

Compound Quantification

Complete calculations should be detailed in this section and should include definitions of terms, dilution factors, calculating to dry or other basis and any other pertinent information. Discussion of actions to be taken if the linear range is exceeded might also be presented here.

Reports

Data Packet Organization

See Appendix II (or whatever) for a check list detailing data packet organization

Quality Control Reports

All results for quality control tests are entered into the lab data base using the QC_DB program. Printouts of all data entered must be included in the data packet. The routine minimum is a method blank report, and an MS/MSD or MS/duplicate report. Many analyses will also require an SRM, blank spike or other report.

Add in any other specific stuff here.

Sample Result Reports

Data Qualifying Flags

Sample report results are qualified with data qualifying flags. These flags have the following definitions:

CODE	Definition
U	: The analyte of interest was not detected, to the limit of detection indicated.

Add any others that pertain to the analysis discussed in this SOP and/or delete this sentence.

Control Chart(s)

The recovery values for list analytes here in the LCS/SRM are plotted on control charts.

Appendix I

Standard Solutions

Appendix II

QC Summary Table

Laucks Testing Laboratories

Method _____ QA Requirements and Corrective Actions

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentati
Initial Calibration					
Initial Calibration Verification					
Continuing Calibration Verification					
MDL standard recovery					
Instrument Blank					
Method Blank					
Surrogate Recovery					
Matrix Spike Recovery					
MS/MSD RPD					
Duplicate % Difference					
Blank Spike Recovery					
Standard Reference Material (SRM) Recovery					

Appendix III

Flow Chart

Appendix IV

(Title here)

Add any other appendices that may be applicable to your method.

Appendix III

Organics Method Template (WORD)

Appendix III

Organics Method Template (WORD)

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LX-????

Title: **(Place title here in bold)**

Revision history:

Number	Date
draft 1.0	08/31/95

Written by: _____ Date: _____
(Author's name typed here & signed above)

Reviewed by: _____ Date: _____
(Supervisor's name & title, delete if not applicable)

Reviewed by: _____ Date: _____
Mike Nelson, Technical Director

Approved by: _____ Date: _____
John Buerger, Laboratory Director

Approved by: _____ Date: _____
Harry Romberg, QC Officer

Title: **(Place title here in bold)**

Operator's signature page

Signature	Printed Name	Date	Read Only	Copy N Assigne

Signature page, for department manager's files

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1. Introduction and Scope

1.1. Method Description

1.1.1. Describe the method, limitations of the method, applicable matrices, target analytes and their detection limits. Also describe where the Laucks method deviates from or makes specific choices defined by the published method on which this procedure is based. Only include variations in the following table. Where Laucks methodology agrees with the published method, it is not necessary to specify.

Method Criteria	Laucks Variations

1.1.2. This method is restricted to use by, or under the supervision of analysts experienced in the use of [gas/liquid] chromatography and in the interpretation of chromatograms. Each analyst performing this method must have demonstrated the ability to perform the described chromatographic analysis and/or data interpretation.

1.2. Sample Collection, Sample Storage, Holding Times

1.2.1. Samples are normally collected in glass containers with Teflon-lined caps. All samples and sample extracts are stored at 4°C. Water samples must be extracted within 7 days of sample collection, soil samples within 14 days of sample collection. All extracts must be analyzed with 40 days of sample preparation. Volatile samples should be analyzed within 14 days of collection. Delete, add to, or make changes in the above where appropriate.

1.3. Definition of Terms

1.3.1. This section defines terms and acronyms as they are used in this SOP. Other terms, such as MS/MSD or method blank, are not defined here since it is assumed that the user of this SOP already understands their more general meaning. This section or parts of it may be deleted if the analysis is basic and has no terminology that may be confusing to anyone likely to read the procedure.

1.3.2. Batch Identifier

A number given to each preparation or analysis group which uniquely identifies that batch. This number is generally the blank ID for preparation batches and either a sequence number for organic analyses or an analysis number which is similar to the blank ID, only preceded by an "A" rather than a "B" for inorganic batches. The preparation batch IDs are discussed in other documentation. You should expand upon how the batch is numbered for your particular application (i.e. B or A, mmddyy, analysis ID, matrix, sequence #).

1.3.3. Blank spike

A background free matrix (DIW for water, clean sand for soils/sediments) to which known amounts of target analytes and surrogates are added each time sample extracts are prepared. Blank spikes are required on all HAZWRAP and NEESA work. In the context of this SOP, a blank spike is the same as a QC check standard. See also QC check standard.

1.3.4. 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.

1.3.5. 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.

1.3.6. 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 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.

1.3.7. Corr Coef, CC

Correlation coefficient. A measure of the "goodness of fit" of a set of data to a regression model. The closer the value is to 1, the higher the degree of confidence in the correlation.

1.3.8. DIW

Deionized water. Lab reagent water. Organic-free water. Since the systems used to provide DIW at Laucks all contain carbon polishing filters, they are capable of providing organic-free water for use in method blanks and method blank spikes.

1.3.9. 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.
1.3.10. ICV	Initial calibration verification. This term is borrowed from the CLP GC/MS protocol. It is a standard which is injected at the start of each QC period that is compared to the initial multi-point calibration to determine whether the instrument is still in calibration.
1.3.11. 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.
1.3.12. 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.
1.3.13. 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.
1.3.14. 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.
1.3.15. 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.
1.3.16. 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.

1.3.17. RRF — Relative Response Factor. A measure of the relative response of an analyte compared to its internal standard. Relative response factors are determined by analysis of standards and are used in the calculation of concentrations of analytes in samples. RRF is determined by the following equation:

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where
A = area of response measured
C = concentration
is = internal standard
x = analyte of interest

1.3.18. 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.

1.3.19. RT, Retention time — The time (in minutes) at which a target analyte elutes from a chromatography column.

1.3.20. RT window — Retention time window. The +/- value which is applied to the ICV to establish the time range used to make tentative compound identifications.

1.3.21. Sequence — A set of sample extracts/digests and standard solutions introduced into an instrument in a chronologically continuous group. See also QC period.

1.3.22. 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.

1.4. Note on Using Spreadsheets for Standard Deviation Calculations

1.4.1. LOTUS 123 computes the population standard deviation when using the @STD( ) function. This value must be multiplied by $\sqrt{\frac{n}{(n-1)}}$ to calculate the correct sample standard

deviation. For 5 data points, this ratio is 1.11. Therefore, the @STD( ) function will underestimate the actual standard deviation by 11%.

1.4.2. If using Quattro Pro, use the @STDS( ) function to calculate the sample standard deviation.

1.4.3. If using Excel, the STDEV ( ) function calculates the sample standard deviation using the correct (n-1) formula.

2. Equipment List and Standards

2.1. Chromatographic System

2.1.1. Describe the chromatographic system, data system, detectors, columns used. Always use "or equivalent" when listing a specific vendor or piece of equipment so that changes can be made without outdating the SOP.

2.2. Standards

2.2.1. Describe the exact preparation of all required standards. Tabulate the concentrations of all analytes in all standards prepared. Include all stock, intermediate, and working solutions, for surrogates, calibration mixes, blank spike mixes, and MS/MSD mixes. Be sure to describe the MDL standard and the IBLK solution.

2.2.2. In Appendix 1, detail all solutions. List all compounds and the concentrations desired. If the list is simple and short and is unlikely to change, it might be best outlined in this section rather than in a remote part of the method. Use whichever approach seems the most appropriate. However, do not neglect this aspect of the method.

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

3.1.5. Grounded power outlets, turning off the instrument and disconnecting the instrument from the electrical power supply before working on any electrical components, etc.

3.1.6. Anything that might be hazardous (working with fire, acid, toxics, etc.) should be listed here and at the point in the procedure where the hazard might exist. Also, delete any part which is not appropriate to the method being written.

3.2. Waste disposal

3.2.1. Describe here the waste(s) generated in using this procedure and the proper disposal methods up to the point of dumping into a waste barrel. etc. Include out of date standards, sample extracts, rinseates, etc.

3.2.2. Waste segregation and disposal from the point of collection is further covered in Laucks SOP LTL-0032.

4. Calibration and Quality Control

4.1. Method Detection Limit Study

4.1.1. Prior to the analysis of any samples, it is necessary to establish method detection limits. This procedure is fully described in Laucks SOP LTL-0027. 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.

4.2. Method Validation

4.2.1. Prior to the analysis of any samples, it is necessary to validate the method. In many cases, the data from the MDL study may be sufficient for method validation. If the RSDs are too high, it will be necessary to perform a method validation study. 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.

4.2.2. The precision of spike recoveries must meet or exceed the criteria tabulated in SW 846. These criteria and recommended spiking concentrations for method validation are tabulated in Appendix IV.

4.3. Retention Time Windows

4.3.1. Prior to the analysis of any samples, it is necessary to establish retention times windows for the method by analyzing standards for all target analytes over at least a 72-hour period. This need not be a single 72 hour sequence but can be 3 chronologically contiguous sequences. These standards should be inter-mixed with real sample extracts in order to mimic actual instrument operating conditions. Tabulate the retention times for all standard compounds and compute the standard deviations of all the retention times. Retention time normalization techniques may be applies if appropriate.

4.3.2. The retention time window half-width is set at 3 times the above calculated standard deviation. This operation must be repeated whenever major equipment changes are made or whenever the chromatographic method is modified.

4.3.3. In some cases, particularly for narrow bore capillary column analyses, the calculated retention time window half widths may be an unrealistically small value or even zero. In such a case, the RT windows may be administratively set. For a complete description of the process of determining RT windows, consult Laucks SOP LX-8000RTW.

4.4. Initial Multi-Point Calibration

4.4.1. Analyze standard solutions using at least 5 different concentration levels. The lowest concentration should be at a concentration near, but above, the method detection limit. The highest concentration should define the upper usable working range of the detector. Inject the standard solutions from the lowest concentration to the highest. Criteria for evaluating these standards should be detailed here. It is likely that only one of the following methods will be used. Delete whatever is inappropriate.

4.5. External Standard Calibration

4.5.1. External standard initial calibration data can be evaluated in one of 3 ways: %RSD of the calibration factors, correlation coefficient of a linear regression analysis, correlation coefficient of a non-linear regression analysis.

4.5.2. CFs are calculated using the equation:

$$CF = \frac{\text{response}}{\text{ng injected}}$$

4.5.3. The distinctions between these methods are: the %RSD method assumes a linear response with the calibration curve passing through the origin, the linear regression method assumes a linear response with a non-zero intercept, and the non-linear method assumes a correlation, but not a linear one.

4.5.4. The calculated CFs are tabulated and the %RSD calculated. Compound-specific criteria are **[List criteria]**.

4.5.5. If the regression method is used, the correlation coefficient must be 0.99 or greater for a 5-point calibration. Note that if this method is used, all CCVs and sample extract concentrations must be computed using the regression equation.

4.5.1. Corrective action

4.5.1.1. If the criteria are not met, the instrument must be re calibrated.

4.6. Internal Standard Calibration

4.6.1. Initial internal standard calibration data are evaluated by determining the % RSD of the Relative Response Factors (RRFs).

4.6.2. RRFs are calculated using the equation:

$$\text{RRF} = \frac{A_s}{A_{is}} \times \frac{C_{is}}{C_s}$$

RRF = Relative Response Factor
A_S = Response of target analyte
A_{IS} = Response of internal standard
C_S = Amount of target analyte, in ng
C_{IS} = Amount of internal standard, in ng

4.6.3. The calculated RRFs are tabulated and the %RSD calculated. Compound-specific criteria are **[List criteria]**.

4.6.4. Note that if this method is used, all CCVs and sample extract concentrations must be computed using the average RRF.

4.6.1. Corrective action

4.6.1.1. If the criteria are not met, the instrument must be re calibrated.

4.7. Initial Calibration Verification

4.7.1. Concentration and/or CF Criteria

4.7.1.1. 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.

4.7.1.2. Using the appropriate calculation technique (average CF, linear calibration, or non-linear calibration, or internal calibration) compute either CFs or concentration values. The calibration factors for the ICV standard are compared to the mean CFs for the initial multi-point calibration. The percent difference for these calibration factors is calculated as follows:

$$\%D = \frac{(C_m - C_i)}{C_m} \times 100$$

where:

C_m = Multi-point average CF
C_i = CF from ICV standard

4.7.1.3. If you are using a regression calibration curve or the internal standard method, calculate concentration values and compute percent difference values:

$$\%D = \frac{(C_t - C_i)}{C_t} \times 100$$

where:

C_t = True concentration
C_i = Measured concentration

4.7.1.4. Compound-specific criteria for the CF method are **[List criteria]**.

4.7.1.5. If the regression or internal standard methods are used, the calculated concentration must be within ??% of the true value.

4.7.1.6. If the regression method is used, the regression curve is used to calculate target analyte concentrations.

4.7.2. Corrective action

4.7.2.1. If the ICV criteria are not met, no sample extracts can be analyzed. Perform system maintenance and re-check the ICV. If the criteria still cannot be met, the system must be recalibrated.

4.8. Chromatographic Resolution

4.8.1. If chromatographic resolution of any compound pairs is an issue, a resolution check must be performed. This check could consist of a separate solution, or could be a part of the calibration mix. The resolution measured must meet the criteria detailed below.

4.8.2. Criteria

4.8.2.1. Detail any resolution criteria here.

4.8.3. Corrective action

4.8.3.1. Perform system maintenance and re analyze the resolution check standard. If satisfactory resolution cannot be demonstrated, no sample extracts can be analyzed.

4.9. Updating Retention Time Windows

4.9.1. The retention time windows for compound identification are updated using the retention times for each target analyte in the ICV standard as the center of the window and the previously determined retention time window half-width to establish the retention time range to be used for compound identification.

4.10. Instrument Blank

4.10.1. Criteria

4.10.1.1. After the analysis of the ICV standard an instrument blank (IBLK) is analyzed. This is to verify that there is no carryover between sample injections. There must be no detectable levels of target analytes in the initial IBLK.

4.10.1.2. Any sample that is suspected of containing high concentrations of target analytes should be followed by an IBLK. This IBLK analysis is used only to make a judgment as to the possibility of carry-over into the sample extract immediately following the IBLK. Evaluation criteria are detailed below.

4.10.1.3. Other IBLKs cannot exhibit a concentration exceeding 10% of the concentration of any target analyte in the preceding sample.

4.10.2. Corrective action

4.10.2.1. If the initial IBLK contains measurable levels of target analytes the system is out of control. The source of contamination must be identified and corrected.

4.10.2.2. IBLKs used to monitor for possible carryover in high concentration extracts (those IBLKs optionally placed into the sequence following suspected high concentration extracts) are used to flag the possibility of analyte carryover into the following sample extract. The extract immediately following the out of control IBLK may need to be re-analyzed if there is a detectable amount of the analyte found in the IBLK.

4.11. Method Detection Limit Standard

4.11.1. Criteria

4.11.1.1. After the analysis of the ICV standard, but before the analysis of any sample extracts an MDL standard is to be analyzed. The MDL standard is used to provide on-going verification of the ability of the chromatographic system to detect analytes at a concentration near the method detection limit.

4.11.1.2. All analytes must be detectable at least 3x signal:noise.

4.11.2. Corrective action

4.11.2.1. Perform system maintenance before analyzing any sample extracts.

4.12. Continuing Calibration Verification

4.12.1. A mid-range calibration standard is analyzed at the frequency of **[Detail frequency here]**. In addition, this standard must be the last injection made in the analysis sequence.

4.12.1. Criteria

4.12.1.1. After every _____ sample extract injections, a CCV standard is analyzed. The CF or concentration for each compound is calculated and the percent difference is calculated as follows. Select the method of choice:

$$\%D = \frac{(CF_i - CF_c) \times 100}{CF_i} \quad \text{or} \quad \%D = \frac{(C_i - C_c) \times 100}{C_i}$$

where:

CF_i = CF from ICV standard

CF_c = CF from CCV standard
or
C_i = Conc. from ICV standard
C_c = Conc. from CCV standard

4.12.1.2. The %D results cannot exceed the detailed CCV criteria, **[List criteria]**.

4.12.1.3. The retention times for all target analytes must fall within the RT windows established by the ICV.

4.12.2. Corrective action

4.12.2.1. Check calculations or perform instrument maintenance. To validate the quantification of target analytes in analytical samples, the samples must be bracketed by in-control CCVs. However, CCV CFs can be outside the control limits as long as the corresponding samples contain no detectable levels of the target analyte for which the CF is out of control, the CF value exceeds the upper control limit (i.e., there is increased sensitivity). Algebraically, this means a greater negative percent difference than the control limit.

4.13. Method Blanks

4.13.1. Criteria

4.13.1.1. Method blanks are used to verify contamination free reagents and apparatus. They are prepared with every set of samples extracted at the same time or one blank every 20 samples which ever is more frequent. Any analyte response above the detection limit is reported. Method blank control limits are detailed below. **[List criteria]** Laucks generally allows no contamination above the MDL for most target analytes. However, blank contamination may be allowed in some circumstances if it is not above certain levels. Specify either the CLP criteria or the SW 846 criteria (< MDL, 5% of the regulatory limit for that analyte, or 5% of the measured concentration in the sample) or whatever the method says.

4.13.2. Corrective action

4.13.2.1. Corrective action may necessitate re-extraction of the sample set. For example if an analyte were found in the blank but not in any of the associated samples then sample group may not require re-extraction. In any event it is the laboratory's responsibility to ensure that method interferences caused by contaminants in solvents, reagents, glassware, and other sample processing hardware leading to discrete artifacts and/or elevated baselines in the chromatograms be minimized. In the extreme case of chronic contamination, blanks

may have to be analyzed from each stage of the sample processing to determine the contamination source so it can be eliminated. In all cases where blank contamination exceeds the control limit a narrative comment must be made which documents the corrective actions taken.

4.14. Method Blank Spikes

4.14.1. Criteria

4.14.1.1. A method blank spike follows the same protocol as with the matrix spike analysis except that the spiking solution is added to a method blank solution instead of an actual sample. A method blank with added analytes is a method blank spike. A method blank spike is the same as a QC check standard. Method blank spike recoveries must meet the criteria specified in the current control limits catalog and/or the quality control database, QC_DB.

4.14.2. Corrective action

4.14.2.1. The method blank spike is used to determine whether a method is in control during sample preparation and analysis. Sample re- extraction and re-analysis would be triggered by an out of control method blank spike only if the sample surrogate recoveries and MS/MSD spike recoveries indicated sample processing errors. If the method does not use surrogates, low blank spike recovery requires that the samples be re-extracted and re-analyzed.

4.15. Matrix Spike

4.15.1. Criteria

4.15.1.1. A sample is chosen at random from the samples to be analyzed, and an aliquot of spiking solution is added to this sample prior to extraction. It is [**if this is an SW 846 procedure,** required that a matrix spike analysis be performed with each extraction batch [**if this is not an SW 846 procedure,** not required that a matrix spike analysis be performed with each extraction batch]. The minimum frequency for MS analysis is 1 each per 20 samples per matrix. This matrix spike sample is used to evaluate the matrix effect of the sample upon recovery of the analytes. The recovery of spike analytes is calculated as follows:

$$\% \text{ recovery} = \frac{(SS-S)}{SS} \times 100$$

where:

SS = concentration in spiked sample
S = native concentration in unspiked sample

4.15.1.2. The recovery criteria are detailed in the Control Limits Catalog and/or the QC database, QC_DB. In the instance that the native target analyte concentration is greater than 5x the spike concentration, the MS recovery control limits do not apply. In this case, treat the MS/MSD pair as duplicates and report them as such in the quality control database (QC_DB).

4.15.2. Corrective action

4.15.2.1. Samples with spike recoveries outside control limits will be reviewed for possible corrective action. Corrective action may involve recalculation, re-extraction, and/or reanalysis. This process should also look at the recovery of surrogate compounds in the MS sample and at the recovery of matrix spiking compounds from the extraction batch blank spike analysis. In all cases a narrative explanation of the condition is required to detail the corrective actions taken.

4.16. Matrix Spike Duplicate

4.16.1. Criteria

4.16.1.1. The compound recovery criteria are identical to those for the matrix spike sample. In addition, the matrix spike duplicate is used 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:

$$\%\,RPD = \frac{|S_1 - S_2|}{(S_1 + S_2)/2} \times 100$$

where:

S_1 = measured concentration for MS sample
S_2 = measured concentration for MSD sample

4.16.1.2. RPD control limits are detailed in the Control Limits Catalog and or the QC database, QC_DB.

4.16.2. Corrective action

4.16.2.1. If a trend in out of control RPD values is observed, the methods used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable reproducibility.

4.17. Sample Duplicate

4.17.1. Criteria

4.17.1.1. At least one duplicate sample per 20 samples per matrix is required. RPD values are calculated in a manner similar to MS/MSD RPDs:

$$\% \text{ RPD} = \frac{|S_1 - S_2|}{(S_1 + S_2)/2} \times 100$$

where:

S_1 = measured concentration in the initial analysis
S_2 = measured concentration in the duplicate analysis

4.17.1.2. The RPD control limits are detailed in the Control Limits Catalog and/or the QC database, QC_DB.

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 Control Limits Catalog and/or the QC database, QC_DB.

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 interferents. 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. Describe the exact conditions for the separation including all temperatures, flows, pump rates, etc.

5.2. Sample Analysis

5.2.1. Analysis sequence

5.2.1.1. See Appendix II for a detailed analysis injection sequence.

5.2.2. Compound Identification

5.2.2.1. Compounds are tentatively identified if a peak elutes in the retention time window characteristic of that compound on the primary column. To confirm the presence of that compound in the sample extract, the peak must also elute in its characteristic retention time window on a second column. Retention time windows are established as previously described and are updated each QC period. Compounds can only be identified if the ICV and CCV criteria 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 ad hoc basis based on such an observed shift. If the data are processed using CHRO, the software allows the operator to increase the retention time window half-width beyond the method- specified width. 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.

5.2.3. Compound Quantification

Target compound concentrations are calculated using the following equations:

5.2.3.1. Aqueous samples

5.2.3.2. The external standard equation, as expressed in SW 846 is:

$$\text{Concentration } (\mu g / L) = \frac{A_x \times A \times V_t \times D}{A_s \times V_i \times V_s}$$

where:

A_x = Response for the analyte in the extract, in area or height units.
A = Amount of standard injected or purged in ng.
A_s = Response for the external standard, same units as A_x.
V_i = Volume of extract injected, µL. For low level purge and trap analysis, $V_i = 1$. For medium level purge and trap analysis there is a V_i term.
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. For low level purge and trap analysis $V_t = 1$. For medium level purge and trap analysis, there is a V_t term.
V_s = Initial sample size, ml.

5.2.3.3. In routine use at Laucks, the equation reduces as follows: First, CF is used directly in the equation. Since CF = A_s/A, this substitution is made. Next, since Laucks routinely measures all final extract volumes in ml, a conversion factor for µL to ml must be made in the numerator of the expression. I.e., µL = 1000 * ml. Finally, the sample preparation process is represented as the algebraic ratio of initial sample size to final effective extract volume, taking into account any intermediate aliquots.

5.2.3.4. The equation then becomes:

$$\text{Concentration } (\mu g/L) = \frac{1000 \times A_x \times D}{CF \times V_i \times (V_s/V_t)}$$

5.2.3.5. This expression is completely equivalent to the SW 846 equation, yielding the same final result. To report concentrations in alternate units, apply an appropriate factor:

$$mg/L = \mu g/L * 0.001$$

5.2.3.6. The equations for internal standard calculations are

$$\text{Concentration } (\mu g/L) = \frac{A_x \times C_{is} \times D}{A_{is} \times RRF \times V_{is}}$$

where A_x, D, and V_s are as in the external standard method.

A_x = Response for the target analyte
A_{is} = Response for the internal standard
RRF = Relative Response factor
C_{is} = Amount of internal standard, in ng

and

$$RRF = \frac{A_s}{A_{is}} \times \frac{C_{is}}{C_s}$$

A_s = Response of target analyte
A_{is} = Response of internal standard
C_s = Amount of target analyte, in ng
C_{is} = Amount of internal standard, in ng

5.2.3.7. Non-aqueous samples

5.2.3.8. The results calculation for non-aqueous samples is very similar to that for aqueous samples. The only difference is the inclusion of a total solids term to calculate the dry weight equivalent of the initial sample size.

$$\text{Concentration } (\mu g/kg) = \frac{1000 \times A_x \times D}{CF \times V_i \times \left(W/V_t\right) \times \left(TS/100\right)}$$

where:

W = Weight of sample extracted or purged, grams.

TS = Total solids, percent.

5.2.3.9. The internal standard calculation for a solid sample is similar to the calculation for an aqueous sample with the inclusion of a term for total solids:

$$\text{Concentration } (\mu g / kg) = \frac{A_s \times C_{is} \times D}{A_{is} \times \text{RRF} \times W_s \times \left(\text{TS}/100\right)}$$

Where W_S = sample size prepared, in gm

6. Reports

6.1. Data Packet Organization

6.1.1. See Appendix III for a check list detailing data packet organization.

6.2. Quality Control Reports

6.2.1. All results for quality control tests are entered into the lab data base using the QC_DB program. Printouts of all data entered must be included in the data packet. The routine minimum is a method blank report, a method blank spike report, and an MS/MSD report.

6.2.2. Add in any other specific stuff here.

6.3. Sample Result Reports

6.3.1. Data Qualifying Flags

Sample report results are qualified with data qualifying flags. These flags have the following definitions:

Code	Definition
6.3.1.1.	The analyte of interest was not detected, to the reporting limit indicated.
6.3.1.2.	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.
6.3.1.3.	The analyte of interest was detected below the practical quantification limit. This value should be regarded as an estimate.

6.3.1.4. The value reported is derived from the analysis of a diluted sample or sample extract.

6.3.1.5. When a dual column /dual detector GC technique is employed, this flag indicates that calculated results from the two determinations differ by more than 25%. Generally, we report the lower value.

6.3.1.6. The value reported is based on a sample or sample extract in which the target analyte concentration exceeded the calibration range. The value reported should be considered an estimate.

6.3.1.7. The target analyte's presence was confirmed by GC/MS.

6.4. Control Chart(s)

6.4.1. The recovery values for list analytes here in the LCS/SRM are plotted on control charts. Corrective action should be employed for instances where the recovery exceeds control limits even once, where recovery exceeds the same warning limit on 3 consecutive occasions, where recovery is on the same side of the mean for more than 8 consecutive points, or where there is any obvious cyclical occurrence or obvious pattern.

7. References

Use the following format:

7.1. *Reference Title*, Author, Publication date

APPENDIX I

Adjust Appendix numbers and titles as necessary for the method being written. Don't forget to do the same in the document references.

Standard Solution Concentrations, units

lowest —> ICV/CCV —> highest

Compound	STD1	STD2	STD3	STD4	STD5	MDL

Calibration Stock Solution, units

Compound	Conc

Surrogate Stock Solution, units

Compound	Conc

IBLK solution, units

Compound	Conc

APPENDIX II

Analysis Sequence

Injection	Sample
	solvent
1	Resolution check standard
2	ICV standard
3	IBLK
4	MDL standard
5	sample

(fill in this table in more detail)

last	CCV standard

APPENDIX III

Data Packet Check List

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, primary column
_____ Chromatographic Report, primary column
_____ Chromatograms, secondary column
_____ Chromatographic Report, secondary column

III. STANDARD DATA:

_____ Initial Calibration Verification
_____ Linearity Report
_____ Surrogate RT Evaluation Report
_____ CCV Reports

Linearity Standards:
_____ Chromatograms, primary column
_____ Chromatographic Report, primary column
_____ Chromatograms, secondary column
_____ Chromatographic Report, secondary column

Continuing Calibration Standards:
_____ Chromatograms, primary column
_____ Chromatographic Report, primary column
_____ Chromatograms, secondary column
_____ Chromatographic Report, secondary column

APPENDIX III, continued

Method Detection Limit Standard:

_____ Chromatograms, primary column
_____ Chromatographic Report, primary column
_____ Chromatograms, secondary column
_____ Chromatographic Report, secondary column

Other Standards Used to Support Sample Data and Instrument Blanks

V. Raw QC Data:

_____ Method Blank
_____ Chromatograms, primary column
_____ Chromatographic Report, primary column
_____ Chromatograms, secondary column
_____ Chromatographic Report, secondary column

_____ Blank Spike
_____ Chromatograms, primary column
_____ Chromatographic Report, primary column
_____ Chromatograms, secondary column
_____ Chromatographic Report, secondary column

_____ Matrix Spike
_____ Chromatograms, primary column
_____ Chromatographic Report, primary column
_____ Chromatograms, secondary column
_____ Chromatographic Report, secondary column

_____ Matrix Spike Duplicate
_____ Chromatograms, primary column
_____ Chromatographic Report, primary column
_____ Chromatograms, secondary column
_____ Chromatographic Report, secondary column

APPENDIX III, continued

V. Bench Sheets

_____ Injection Sequence
_____ CHRO Sequence
_____ LAS Method
_____ CHRO Method
_____ Extraction Bench Sheets
_____ Miscellaneous Work Sheets. ie. %TS, SDG summary, calculations, HTVR
_____ Standards Logs

VI. Reject Data:

DO NOT COPY DO NOT PAGINATE

Data not used to support sample results.
All data acquired but rejected on account of QC out of control.
Non-routine standards used to support sample data should be placed at the last of the Standard Data section.

APPENDIX IV

Method Validation Criteria

Compound Criterion	Recommended Concentration	RSD

APPENDIX V

QC Summary Table

Laucks Testing Laboratories

Method _____ QA Requirements and Corrective Actions

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentatic
Tuning					
Initial Calibration					
Initial Calibration Verification					
Continuing Calibration Verification					
MDL standard recovery					
Instrument Blank					
Method Blank					
Surrogate Recovery					
Matrix Spike Recovery					
MS/MSD RPD					
Duplicate % Difference					
Blank Spike Recovery					
Standard Reference Material (SRM) Recovery					

APPENDIX VI

Method Flow Chart:

Create a flow chart using "Flow Charting 4" or equivalent. This can then be imported directly into this SOP template. Keep the number of pages to a minimum, one page if possible.

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1002

Title: **Document Tracking and Control**

Revision history:

Number	Date
1.0	05/15/95
2.0	12/27/95
3.0	1/06/98

Written by: Harry Romberg, QA Officer Date: 1/6/98

Approved by: Karen J. Kotz, Laboratory Director Date: 1/9/98

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SOP UPDATE FORM 17

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 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 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 and Updating of Documents

2.1.1 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 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.

2.1.2 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.

2.1.3 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. 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. These forms will also be issued approximately annually in order to initiate the review process for existing SOPs.

2.1.4 It is recognized that some documents may have been written prior to completion of the Document Control Form or that it may be decided that some documents which are already in existence should be placed into the document control system. Unless these documents are ready for immediate approval, and acceptance by the Lab Director, QA Department and/or other responsible parties, in other words, not in a draft or review status, the document control form should be filled out.

2.1.5 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.**

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. The scheme for numbering 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-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
-7400	Flame Atomic Absorption Analyses
-7500	Cold Vapor Atomic Absorption Analyses
-7600	Gaseous Hydride Atomic Absorption Analyses
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.

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."

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 are optional. 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.

SOP No: LTL-xxxx
Revision: 1
Date: 04/13/98
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 required 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 the most recent revision date, the next revision due date, the last review date and, if any version existed under the previous SOP system, the previous SOP number. Only the applicable fields among these latter fields need be filled out. A copy of the screen form is presented in Appendix B.

2.2.8 Types of reports available from the SOP database include a table of contents for each SOP book, which are printed out whenever SOPs are released, automated Document Control Forms similar in content to the one in Appendix A, reports on SOPs due for review, and reports on SOPs overdue for review. The latter forms enable QA to assign and to track the status of SOPs which are up for their annual review. As this is an Access Database, any other type of query or report form can be generated that uses any of the information previously noted in the above paragraph and in Appendix B.

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 either 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 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. In addition, the SOP book table of contents will be updated to reflect the revised SOP(s).

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

2.2.12 When documents are distributed to the departmental supervisor, a copy of the signature list(s) for the specific document(s) is/are also distributed. The signature lists are returned to the QA department when completed.

2.2.13 Departmental supervisors will insure that the most recent version 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 signature lists for the current document are signed and dated as the staff complete reading the SOPs. In addition, as staff new to a particular task (SOP) are trained, the departmental supervisor will insure that they have read and signed the signature list for that SOP. This may require that the supervisor request a new SOP signature list for that staff member so that they can sign the SOP for newly assigned tasks.

- The departmental supervisor is responsible for insuring that all outdated versions of SOPs are discarded or destroyed when the newer revisions are issued.

2.2.14 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.

2.2.15 Note that versions of methods or SOPs which have been given to regulatory agencies or clients are uncontrolled in that they will not be updated except by specific arrangement.

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. 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 .R0 or .R1, 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. No other copies of these controlled documents should be kept by laboratory staff in order that unapproved copies of the document do not proliferate.

2.4 Review and Updating of Documents

2.4.1 In order to facilitate updates to documents without violating the practices outlined in the SOP, and in order to insure all approved updates have indeed been incorporated into the document, an "SOP Update" form (Appendix E) should be used. This form may be filled

out at any time by an analyst or supervisor. Before the change can be brought into practice, however, it must approved by QA. QA may also choose to consult the area supervisor, Division Manager, or other senior staff before incorporating the procedure into the routine practice. A copy of this form will be kept with the laboratory controlled copy **AND** a copy must be filed with QA. When it is time to update the SOP, changes outlined on these forms will be incorporated into the revision.

2.4.2 Unless major changes to SOPs are required, SOPs should be reviewed approximately annually. Changes which do not require immediate update are typos or wording changes which do not inhibit the correct interpretation of the operation involved. Items which could lead to misinterpretation or incorrect performance of the methods should be corrected as soon as feasible. At the time of any revision, items addressed in the "SOP Update" forms will be incorporated into the SOP. In addition, any other updates determined at the time of the review will be added. Each review will be documented on the Document Control Form (Appendix A).

Appendix A

Document Control Form

Laucks Testing Laboratories

DOCUMENT CONTROL FORM

_____ Generate new document

_____ Modify existing document

_____ Review existing document

Document No.: _____

Document Title: _____

_____

_____

Assigned to: _____ Date: _____

The aforementioned document has been reviewed and does not require modification at this time:

Reviewer: _____ Date: _____

Purpose for generation or modification of document and comments on review:

_____

_____

_____

_____

_____

_____

QA Approval: _____ Date _____

Appendix B

SOP Database Screen

Use the [Page Up] and [Page Down] keys to browse through the Document List.

Search for a record | Go to top of DOC. List | Open a blank record | Update Assignment List | Delete this record | Close Doc. List Window

Current Document #: LTL-1002

Revision Date: 12/27/95

CHOOSE Department: Quality Assurance

Next Revision Due: 7/15/96

Revision #: 002

Last Review Date: 12/27/95

CHOOSE Supervisor Name: Romberg, Harry .

Previous Document #: LTL-0054

Document Name: Document Tracking and Control.

Department Abbreviation: QA

Current SOP #: LTL-1002

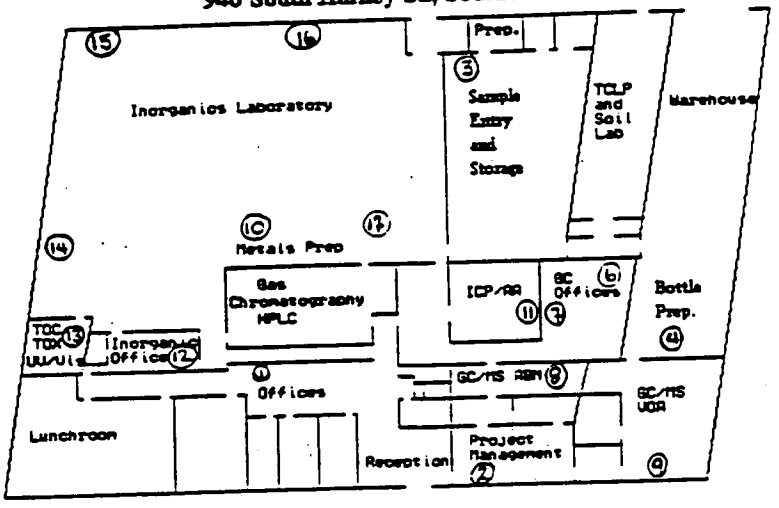
SOP Distribute: Yes

Record: 1 of 13

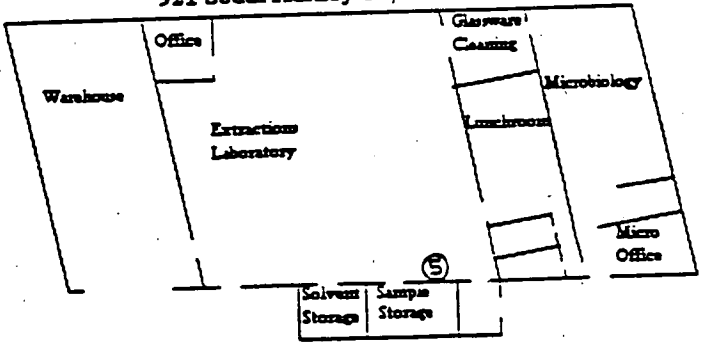
Appendix C

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

Document Control Stamp

Controlled Document

No. _____ Assigned to: _____

Appendix E

SOP Update Form

Laucks Testing Laboratories

SOP UPDATE FORM

Document No.: _____

Document Title: _____

_____

_____

The following changes have been reviewed and determined to be necessary to the implementation of the above document.

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

Submitted by: _____ Date: _____

Approved by (QA): _____ 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

Written by: Harry Romberg Date: 2-3-98
Harry Romberg, Quality Assurance Officer

Approved by: Karen J. Kotz Date: 2/3/98
Karen Kotz, Laboratory Director

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APPENDIX A 8

SECURE STORAGE CUSTODY LOG 8

APPENDIX B 10

VOLATILES CUSTODY LOGS 10

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.

1.2 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 logs, either generated by the client or by Laucks. Often these chains-of-custody are intended only for clear identification of testing parameters, rather than actual custody maintenance. These custody logs, however, will always be signed, timed and dated by the person checking the samples in and entering them into the laboratory database.

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 require 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 HAZWRAP, NFESC, or Army Corps of Engineers 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 3 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.
- The volatiles refrigerators are located in the area between the GC room and the laboratory computer system hub.
- 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** 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 Numbers**" column should indicate which samples are being removed for analysis (i.e. 1-10 metals digestion, or 3-5 NO3/NO2 analysis).
- 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 Numbers**" column should indicate which samples 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 Numbers**" column should indicate which samples 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 must 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, the 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 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 Number	Matrix (*optional*)	Location (*shelf*)	Logged Out		By	Logged In		By	Action
			Date	Time		Date	Time		

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

GC Volatiles Custody Log
Laucks Testing Laboratories, Seattle, Washington

		Date/Time		Date/Time		
Samples	Analysis	Removed	Analyst	Returned	Analyst	Comment

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1004

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

Revision history:

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Revised by: Harry Romberg Date: 6-23-96
Harry Romberg, Quality Assurance Officer

Approved by: Karen J. Kotz Date: 6/23/96
Karen Kotz, Laboratory Director

UNCONTROLLED

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1. Introduction and Scope

1.1 Description

1.1.1 This SOP describes the way in which analyst competence is initially documented and by which the analyst is considered capable to perform independent analysis. Two practices are in place at the time of this writing. One practice is designed primarily for analysts who have been employed doing an analysis for a significant period of time at Laucks and have demonstrated competence through the successful analysis of many samples, including one or more of the following: performance evaluation (PE) samples, reference materials, laboratory control samples, surrogates, etc. The other practice is primarily for analysts who have been performing a specific analysis for less time than is considered extended proof of competence. This practice involves the analysis of multiple aliquots of a PE sample and subsequent evaluation of the results.

1.2 *Scope*

1.2.1 *This SOP contains discussion of initial demonstration of competence through PE analysis and, for some analyses, P&A criteria. It also defines ongoing performance demonstration through the use of PE samples.*

1.2.2 *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** - Performance Evaluation*
- ***P&A** - Precision and Accuracy*
- ***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.*

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 competency items outlined in this SOP in a manner consistent with the analytical SOP.*

3.1.3 *The analyst must analyze a PE study initially and on an ongoing basis (at least annually) for each method for which they are considered qualified.*

3.1.4 *For many analyses, the analyst must perform an initial Precision and Accuracy study as required.*

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.*

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 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.*

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.*

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

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).*

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

3.3.3 *QA reviews PE and P&A studies to ensure criteria have been met.*

3.3.4 *QA works with managers to assist in developing training materials.*

3.3.5 *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 Recognition of Experience and Training

4.1.1 Many analysts have been performing their assigned duties for an extended period of time and have successfully analyzed many samples, reference materials, PE samples, matrix, blank, and surrogate spikes and have not only demonstrated their capabilities to achieve results which meet criteria but have demonstrated a thorough knowledge of all aspects of the chemistry involved, instrument performance and maintenance, the necessary data reduction requirements, quality control criteria, and documentation.

4.1.2 These analysts, at the discretion of the appropriate Division Manger, may be certified to independently perform their analytical duties. This is achieved using the Recognition of Experience and Training Form, an example of which is in Appendix A. This form contains space to note the analysis type (Cyanide, for example) and the methods by which they are considered competent (335.3 and 9012 perhaps, but not CLP). The dates from which they have been doing these analyses must also be noted on the form. The Division Manager then signs the form in order to certify that the analyst is considered adequately trained in the particular method or aspect of the job. The form must include the criteria used to designate someone as competent and attached to the form must be the applicable documentation to confirm the criteria has been met.

4.1.3 *Certification of competency must include the successful analysis of a performance evaluation (PE) sample where such are available or can be made in the laboratory by a supervisor. This sample will be blind to the analyst, must be analyzed independently by them and must be analyzed in accordance with the appropriate SOP. Greater specifics on these types of samples are given in the Laucks SOP entitled "Blind Spike Program" but will often be from a WP or WS study or from another commercial source. Analysts who have been performing analyses for any length of time at Laucks have almost certainly analyzed numerous PE samples which can be used for initial and ongoing demonstration of competency.*

4.1.3.1 *Adequate performance on a PE sample will be considered to be within the supplied statistical limits for that sample if from a commercial source or from method defined limits for an LCS or blank spike if from internally prepared material.*

4.1.4 *Precision and Accuracy (P&A) criteria using quadruplicate analysis are also a part of most organic SW846 and some other methods. Successful analysis of such samples will be considered to be within the reference method-specified criteria. Since Laucks own precision and accuracy limits must be within the method-specified criteria, the analyst should also be able to meet Laucks criteria as well as those of the reference method. However, as long as method criteria are met, the analyst may be approved for independent work as long as they are able to obtain satisfactory performance from the ongoing analytical QC for that analysis.*

4.1.5 It is acceptable to certify such capabilities on multiple forms and to certify for multiple analysis types and/or methods on one form. At the time of this writing, there may be no known materials which can be submitted as unknowns for some analyses. In this event, at the discretion of the Division Manager and Quality Assurance Officer, this form may also be used to qualify analysts. From the date of the first version of this SOP, however, this should not be done where materials are readily available and reasonably handled.

4.1.6 When this process is completed, the original of this form and a copy of all applicable documentation will be inserted into the analyst's training file which is maintained in the QA area for the 940 building and the Extractions Supervisor Office for the 921 building.

4.2 Demonstration of Capability to Perform Analysis

4.2.1 For analysts who are relatively new to their assigned tasks, *a greater degree of* capability *demonstration must* be *undertaken* through *the satisfactory completion of any internal departmental training documentation. This training will include specific training and documentation developed by that department and department manager and may include required reading, quizzes, and performance criteria at the discretion of the department manager and QA. Example checklists are provided as Appendix C.*

4.2.2 *In general, if an analyst has not passed the criteria detailed in 4.1, then he/she must proceed through the following:*

4.2.2.1 *A trainer is designated for the task/test*

4.2.2.2 *One-on-one training occurs for the timeframe designated by the supervisor and applicable checklists.*

4.2.2.3 *Training may also include required reading of SOPs and the QA Plan, quizzes, and subset task demonstrations.*

4.2.2.4 *Progress is monitored and documented on applicable forms.*

4.2.2.5 *Supervised training continues until the analyst is deemed ready for capability demonstration.*

4.2.2.6 *Demonstration of analytical competency completion, however, will be the same. Performance Evaluation and/or P&A elements as described previously in 4.1.3 and 4.1.4.*

4.2.3 *Where P&A demonstration is not required and defined by the method, Laucks may choose to apply additional internal P&A criteria similar to a typical P&A study.* The samples may be submitted by the QC Officer, the Division Manager, or an individual designated by one of the above. Four or more aliquots of a material will be submitted to the analyst as unknowns. The analyst must demonstrate the capability to achieve results within the recovery range specified by the manufacturer, if they are independent materials, or within laboratory recovery criteria if they are prepared in-house. In addition, the % RSD of the results must be within Laucks established RPD limits (or default RPDs if none exist for a specific target analyte).

4.2.4 It is recognized that some independent materials may not recover within manufacturers criteria, at least for a subset of the target analyte list, regardless of the experience and competence of the analyst, due to degradation of the material, arbitrary setting of the limits, determination of the "true" values by methods other than those used for the analysis, or other factors. In that case, the % RSD may be the major factor in evaluation and other considerations or action may be taken at the discretion of the QC Officer and/or Division Manager, such as how Laucks more experienced analysts have historically performed for a particular material.

4.2.5 Failure to meet criteria means that the analyst must continue to work under the close supervision of a trained analyst.

4.2.6 Likewise, meeting these criteria may be determined to be only one step in the overall training process. Whereas this is demonstration that the analyst is capable of obtaining reliable results, the Division Manager or other supervisory personnel may determine that a more complete knowledge of the analytical process is in order, such as instrument maintenance capabilities, method troubleshooting, data reduction, proven performance on actual sample analysis, etc.

4.2.7 When such materials are analyzed, a Demonstration of Capability to Perform Analysis form is completed (see Appendix B). This form is designed for single analyte methods. For multi-analyte materials, a page may be attached which depicts all of the analyst's results and the control criteria. However, this is the final signature form and must accompany any summary pages or written evaluation which may be considered pertinent. Also attached should be copies of the supporting data or a data summary page which references the workorder under which the data may be found.

4.2.8 The date of analysis, the results, the recoveries, and the % RSD are recorded on the form (or the attached summary). If all analytes met or did not meet criteria, the appropriate box is checked. If not all criteria are met but the analyst was considered to have performed adequately, a narrative explanation *must* accompany the evaluation, either on the back of the form or as a separate, attached report.

4.2.9 Additionally, if the analyst, through the analysis of these samples is considered fully qualified to perform the analysis, the appropriate box is checked and the form signed by the Division Manager. If the Division Manager considers that the analyst is now capable of analysis but still requires additional experience and training before they are fully capable of independent analysis, a date is set to review performance. The additional experience or training required and the next performance review date are recorded on the form (with the appropriate box checked) and initialed.

4.2.10 If further training is still required, copies of these forms will be retained by QA in a file to be reviewed regularly to insure that this final analyst review occurs in a timely fashion. A copy of the form indicating interim status will also be retained in the staff member's training file.

4.2.11 When this process is completed, the original of this form will be inserted into the analyst's training files.

4.3 *Ongoing Demonstration of Performance*

4.3.1 *At least annually, after initial qualification, analyst proficiency must be demonstrated. Each staff member that performs a method must demonstrate their continued proficiency through analysis of single blind proficiency samples (another PE). WP, WS or*

commercial PE samples may be used to satisfy this requirement just as they were used for initial qualification.

4.3.2 *As with initial qualification, continuing performance must be documented in the analyst's training file. Ongoing competency can be documented using the Recognition of Experience and Training Form.*

5. *References*

Navy Installation Restoration Laboratory Quality Assurance Guide, Naval Facilities Engineering Service Center, February 1996

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

Appendix A

Recognition of Experience and Training Form

Recognition of Experience & Training Form

Laucks Testing Laboratories

It is hereby recognized that _____
Employee Name

has demonstrated competence in the methodologies listed below. Through the successful analysis of numerous samples, including performance evaluation samples, matrix spikes, laboratory control samples, etc. and in the associated reduction of data as required by these methods, we certify this staff member as being capable of independent performance of the listed analyses.

Analysis Type	Method Numbers	Has Been Performing Analyses by These Methods Since	Has Demonstrated Competency by meeting the following criteria, with the hard copy of applicable information relating to this competency attached to this form

_____
Division Manager

_____
Date

Appendix B

Demonstration of Capability to Perform Analysis Form

Demonstration of Capability to Perform Analysis

Laucks Testing Laboratories

Analyst:_____

The above analyst has independently analyzed at least 4 aliquots of the listed performance evaluation material, which were submitted as blind samples, achieving the listed recoveries. The limits specified by the manufacturer are considered within acceptable range or, if prepared by Laucks from known materials, the laboratory established control limits apply. In addition, the % relative standard deviation (%RSD) of these data is evaluated against the laboratory established RPD limits as set at the time of this evaluation.

Method:_____ PE Material:_____

Target Value:_____ Recovery Criteria:_____

Reproducibility Criterion:_____

Date	Result	% Recovery
%RSD:		

Criteria for non-analytical functions:_____
Demonstrated by:_____

☐ Met Criteria ☐ Did Not Meet Criteria

These data are considered adequate demonstration of independent performance if all criteria are met. Other factors may prevail, at the discretion of the appropriate Division Manager before any analyst may be allowed to independently analyze actual samples.

☐ Analyst has met performance criteria but requires more experience. Specific areas which require further training or experience are _____ Work will be reviewed in _____ and capabilities evaluated. *[Initial here. Do not sign below]*

☐ Analyst has met performance criteria and has been found fully capable of independent work. *[Sign Below]*

_____ Division Manager

_____ Date

Appendix C

Example Training Checklists

Laucks Testing Labs

Pesticide/Herbicide GC Semivolatile Analyst Training Verification Checklist

Analyst Name:		Date:	Trainer:	Supervisor:	Analys
cumentation					
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 Methods (SW846, CLP, 500 & 600 series)				
	List Method(s):				
6	Has read and understands appropriate sections of GC Training Manual				
Instrument Operation/Maintenance					
7	Knows location and use of Instrument Manuals				
8	Knows basic GC theory				
9	Able to use GC Control Pad to set temperature program				
10	Able to use Autosampler Control Pad to set injection program				
11	Able to change syringe, septa & injection port liner				
12	Able to trim/change columns, install Y connector & perform leak check				
13	Able to measure and set carrier and makeup gas flows				
14	Able to bake column/injectors/detectors				
15	NON-ROUTINE: *Able to change detectors*				
16	NON-ROUTINE: *Able to perform total system cleaning*				
nalytical Performance					
	Able to prepare standards & pass standard QC acceptance criteria				
18	Able to analyze breakdown check and apply 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 surrogates and spikes				
22	Able to set up analytical runs (CLP & non-CLP) & acquire data				
23	Able to get information on samples/analyses (test codes, MDLs, etc.)				
24	Able to quantitate an analytical batch (standards, CCVs, QC & samples)				
25	Knows how to confirm detection of analytes (peak ID, conf. col.)				
26	Knows reanalysis and reextraction criteria				
27	Able to perform sample dilutions (obtaining linear results)				
28	Knows correct reporting limits for method(s)				
29	Knows corrective action & documentation for out of control QC events				
30	Able to p. duce a data package (In-house, CLP and SW-846)				
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 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
HPLC Semivolatile 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 Methods (SW846)				
	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 checks				
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 Helium tank				
17	NON-ROUTINE: *Able to change lamps*				
18	NON-ROUTINE: *Able to locate the high pressure build-up*				
19	NON-ROUTINE: *Able to change pump seal*				
20	NON-ROUTINE: *Able to clean flow cell*				
Analytical Performance					
21	Able to prepare standards & pass standard QC acceptance criteria				
22	Able to analyze and generate acceptable calibration curve				
23	Able to analyze CCVs and apply QC acceptance criteria				
24	Applies acceptance criteria for surrogates and spikes				
25	Able to set up analytical runs & acquire data				
26	Able to get information on samples/analyses (test codes, MDLs, etc.)				
27	Able to quantitate an analytical batch (standards, CCVs, QC & samples)				
28	Knows how to confirm detection of analytes (peak ID, conf. col.)				
29	Knows reanalysis and reextraction criteria				
30	Able to perform sample dilutions (obtaining linear results)				
31	Knows correct reporting limits for method(s)				
32	Knows corrective action & documentation for out of control QC events				
33	Able to produce a data package (In-house and SW-846)				
Method Validation *(complete one or more of the following)*					
34	Has successfully analyzed four P&A samples				
35	Has successfully analyzed two PE samples				
36	Has successfully analyzed three each of two types of QC samples				

This is to certify that _____ has been an analyst in the HPLC semivolatile 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

Semivolatile Analyst Training Verification Checklist

This is to certify that _____ has been an analyst in the semivolatile GC/MS department and has demonstrated competency at the following tasks:

	Task:	Date:	Supervisor:	Analyst:
1	Able to Log-on to the RTE system			
2	Able to create and edit BLISTS			
3	Able to create spectra and quant reports			
4	Able to prepare sample extracts for analysis (including dilutions)			
5	Able to do basic mass-spec tuning			
6	Able to perform daily maintenance tasks			
7	Able to change a helium tank			
8	Able to enter data into SAM special tests and QC reports			
9	Able to get a basic directory listing of files on the RTE			
10	Able to use QAREA			
11	Able to check a CCV standard for compliance to the method			
12	Able to check DFTPP for compliance to the method			
13	Able to use basic RTE EDIT commands (create and edit files)			
14	Able to use basic RPN commands (EC, DR, PF, PBM, etc.)			
	Able to generate simple TIC data			
	Able to check spectra vs. standard spectra			
17	Applies acceptance criteria for surrogates, spikes, & ISs			
18	Knows the basic differences between In-House, CLP, and SW-846			
19	Able to generate basic CHRO forms packages			
20	Knows where to get information on samples (test codes, etc.)			
21	Able to calculate RFs and results from raw data			
22	Knows the types of extraction procedures used for ABNs			
23	Knows basic GC/MS theory			
24	Has read and understands the SOPs for all applicable methods			
25	Knows corrective action for out of control QC events			
26				
27				
28				
29				

Work Order:	TICs?	Package SDG.	TICs?

GC/MS Semivolatile Analyst Competency Criteria

	Task:	Criteria:
1	Able to Log-on to the RTE system	Observation
2	Able to create and edit BLISTS	Successfully create 4 BLISTs
3	Able to create spectra and quant reports	Done for 4 jobs
4	Able to prepare sample extracts for analysis (including dilutions)	Done for 4 jobs
5	Able to do basic mass-spec tuning	Submit 4 tune checks / observation
6	Able to perform daily maintenance tasks	Observation
7	Able to change a helium tank	Observation
8	Able to enter data into SAM special tests and QC reports	Done for 4 jobs
9	Able to get a basic directory listing of files on the RTE	Observation
10	Able to use QAREA	Observation
11	Able to check a CCV standard for compliance to the method	Observation
12	Able to check DFTPP for compliance to the method	Submit 4 tune checks
13	Able to use basic RTE EDIT commands (create and edit files)	Observation
14	Able to use basic RPN commands (EC, DR, PF, PBM, etc.)	Pass RTE quiz at 85%
15	Able to generate simple TIC data	Done for 4 jobs
16	Able to check spectra vs. standard spectra	Done for 4 jobs
17	Applies acceptance criteria for surrogates, spikes, & ISs	Done for 4 jobs
18	Knows the basic differences between In-House, CLP, and SW-846	Observation
9	Able to generate basic CHRO forms packages	Complete 2 packages w/o supervision
20	Knows where to get information on samples (test codes, etc.)	Observation
21	Able to calculate RFs and results from raw data	Correctly complete 4 examples
22	Knows the types of extraction procedures used for ABNs	Observation
23	Knows basic GC/MS theory	Has read training manual
24	Has read and understands SOPs for all methods	Observation
25	Knows corrective action for out of control QC event	Observation / has read SOPs
26		
27		
28		
29		

GC/MS Training Program

Criteria for Demonstration of Analytical Competency

Analyst Name: _____

The analyst must meet at least one of the following criteria to demonstrate analytical competency.

1. Successfully analyze four (4) precision and accuracy samples, which have been prepared according to the SW-846 criteria for the method validation, or, if this is not available, according to in-house criteria. The results must be within limits specified by the SW-846 method or, if unavailable, by in-house protocol. (Attach data to this sheet).

Completed on : _____ Supervisor: _____

2. Successfully analyze two (2) rounds of performance evaluation samples. The results must be "acceptable" for 90% of the total compounds analyzed in multi-compound methods. If two rounds of samples are not available within six (6) months, one round of PE samples and the criteria from section 3 below will be acceptable. (Attach data to this sheet).

Completed on : _____ Supervisor: _____

3. Successfully analyze three (3) each of any two (2) of the following QC samples (total of 6 QC sample results). The results must be within the control limits for all compounds analyzed. (Attach data to this sheet).

MS/MSD _____
SRM _____
Blank Spike _____

Completed on : _____ Supervisor: _____

In addition to analytical competency, non-analytical competency must be demonstrated by the criteria found on the Semivolatile Analyst 3rd Month Training Verification Checklist.

REPORT MANAGEMENT

Employee Name: Date of Hire:

ACTIVITY Reporting	Approx. Training Schedule	SOP Number (where applicable)	Trainer Initials/Date	Trainee Initials/Date
Transcribing data from sub labs				
Reporting– Extractions Metals Semi-volatiles Volatiles				
Log-in new project folders				
Log-out complete folders				
Update SAM reports/benchsheets				
Cover letters				
Faxing				
Logging data in folders				
Filing benchsheets/WOC forms				
Filing complete folders				
Archiving complete folders				
Updating Paradox files				
Copying reports				
Paginating reports				

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
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Revised by: Harry Romberg, QA Officer Date: 5-13-98

Approved by: Kathy Kreps, Laboratory Director Date: 5-18-98

Controlled Document

No. 20 Assigned to: Tetra

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1. Introduction and Scope

1.1 General

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

1.1.2 The purpose of this SOP is to insure the proper use and calibration of all analytical balances in the laboratory. It involves the daily use of 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, analytical balances are cleaned and general maintenance 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. 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 the calibrations will be suspended or other Class "S" weights used (if available) until the calibrated weights return.

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 <u>clean</u> before use.
- The balance is <u>level</u> before use.
- The balance is <u>clean</u> and <u>level</u> 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.
- **<u>Prior to use, the user should insure that the daily calibration check has been done</u>**. 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

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 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
May 4 - May 7, 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
OK	50.0	50.00		OK	OK	OK	OK	OK
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				
OK	50.0	50.00		OK				
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

Written by: Harry Romberg Date: 3-24-98
Harry Romberg, QA Officer

Approved by: Kathy Kreps Date: 3/24/98
Kathy Kreps, Laboratory Director

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1. Introduction and Scope

1.1 Method Description

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

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

2. Equipment List

2.1 Equipment

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

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 a 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 The NIST traceable standard thermometer is recalibrated annually by sending it back to a manufacturer who has the capability to recalibrate thermometers to NIST specifications. Currently, Laucks uses the EverReady Thermometer Company (ERTCO) 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 know his/her 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.

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

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 A). 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 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 boiling water bath. The standard and individual thermometers are inserted into a beaker of boiling water up to the immersion line. 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 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 to the individual thermometer reading will result in a "correct" temperature.

5.3.4 Correction factors may be written on the thermometer 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 The standard thermometer is placed in a glass Erlenmeyer flask filled with water in a cold storage unit at least overnight (12 hours).

5.4.2 The infrared thermometer is used to measure the temperature of the flask while it contains the standard thermometer.

5.4.3 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.

5.4.4 This emissivity setting, 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 a glass container and use the emissivity setting noted on the IR thermometer in order to get an accurate temperature measurement.

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

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 corrective action as noted in this SOP and on the monitoring form or to see that corrective action is initiated by informing a supervisor. 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 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.

7.4.2 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 than 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. 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 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
1997 Laboratory Thermometer Calibration

Cold Storage ID	Location	Serial No.	Cal. Date	S/N-FC-53631 NIST reading	Thermom. Reading	Correction Factor
C01	Inorganics	ERTCO 4156	6/6/97	5.0	6.0	-1.0
C02	GC residues	none	6/6/97	5.0	5.4	-0.4
C03	extractions	B B01679	6/6/97	5.0	6.1	-1.1
C04	GC VOA	B B03987	6/6/97	5.0	5.5	-0.5
C05	Inorganics	B02580	6/6/97	5.0	5.2	-0.2
C06	Warehouse	B B04765	6/6/97	5.0	5.8	-0.8
R02	GC Semi Stds.	B B03534	6/6/97	5.0	5.7	-0.7
R04	GC/MS whse.	B B00906	6/6/97	5.0	5.3	-0.3
R06	extractions	B B01708	6/6/97	5.0	5.6	-0.6
R07	extractions	B B04405	6/6/97	5.0	6.2	-1.2
R08	Inorganics	B03021	6/6/97	5.0	5.5	-0.5
R11	929 warehouse	B B01342	6/6/97	5.0	6.0	-1.0
W01	940 Walk-in	B B03928	6/6/97	5.0	5.4	-0.4
W02	921 Walk-in	B B01919	6/6/97	5.0	5.8	-0.8
F03	GC semivolatiles	B B B08959	6/9/97	-15.6	-16.2	0.6
F04	GC semivolatiles	B B B09215	6/11/97	-15.7	-15.6	-0.1
F05	GC/MS VOA	B B B06608	6/9/97	-15.6	-16.5	0.9
F06	extractions	B B B08543	6/9/97	-15.6	-15.8	0.2
F07	GC/VOA stds.	B B B08765	6/9/97	-15.6	-16.1	0.5
		Ertco 5236	6/9/97	-15.6	-17.0	1.4
		Ertco 4268	6/9/97	-15.6	-15.8	0.2
		Ertco 5034	6/9/97	-15.6	-15.6	0.0
		B B04059	6/10/97	5.4	5.4	0.0
B B B09215 had a broken Hg column and registered +4 degrees high. The thermometer was repaired and returned to service.						
IR Thermometer (ITT-330)		Horiba 226099	6/13/97	4.5	6.0 (at E=85)	
			6/13/97	4.8	4.8 (at E=80)	

Laucks Testing Laboratories
1997 Laboratory Thermometer Calibration

Oven or Use		Serial No. or ID No.	Cal. Date	NIST read	Thermom. Reading	Correction Factor
VWR 1320 (replaced w/ 61019-204)		1A	6/18/97	101.4	100.0	1.4
Thelco		2	6/18/97	101.2	101.5	-0.3
VWR 1310		3	6/18/97	101.2	100.1	1.1
VWR 1330	#4 Univ. Enterprises	L12-004	6/18/97	101.2	100.9	0.3
VWR 1370		5	6/18/97	101.2	101.2	0.0
VWR 1330 (Extractions)		7	6/18/97	101.3	100.0	1.3
Blue M (Extractions)		8	6/18/97	101.3	103.8	-2.5
back waterbath (ASTM 1F)		VWR 02429	6/18/97	101.2 C	213.5 F	0.7 F
front waterbath		(Made in UK)	6/18/97	100.3	99.9	0.4
1300 U (TOC room)		SPER Sci. 106	6/18/97	97.0	97.0	0.0
Pensky Martin (ASTM 9F)		VWR 61091-001	6/18/97	100 C	212 F	0.0 F
BOD thermometer		1	6/24/97	20.7	20.8	-0.1
Napco Oven		SPER Sci.	6/25/97	101.2	100.5	0.7
Digestion Area		Polyscience USA	6/25/97	101.2	103.6	-2.4
VWR 1320		61019-204	6/25/97	101.2	102.0	-0.8

Appendix II

Example Cold Storage Temperature Log

Cold Storage Temperature Log

Laucks Testing Laboratories, Inc.

Cold Storage ID #:_____ Thermometer ID:_____

Location:_____ Year: **1998** 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												

Q:\TEMPLATS\LTLFORMS\REFERFRM.DOC 3/19/98

Record Time and Temperature in the proper blocks and initial the entry each day of normal laboratory operation.

If refrigerator temperatures exceed 4°C±2°C or if freezer temperatures are warmer than -10°C, corrective action must be taken.

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 ac[illegible] **MUST** be recorded on this log sheet. If there is insufficient room, mark on the back of [illegible]ge with the date the action occurred.

Samples MUS[illegible]T be stored in units which are not maintaining the proper temperature.

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

Revised by: Harry Romberg, QA Officer Date: 5-13-98

Approved by: Kathy Kreps, Laboratory Director Date: 5-18-98

Controlled Document

No. 20 Assigned to: Tetra

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1. Introduction and Scope

1.1 Introduction

1.1.1 The maintenance of instrument logbooks is essential to monitoring instrument performance and throughput and in tracking analyses. It is also important to confirming instrument performance at the time of specific analyses and in monitoring ongoing or periodic performance degradation and the steps taken to correct or prevent such occurrences. Several systems are in place at Laucks, the differences being primarily dependent on the specific instrument and analysis types. This SOP will discuss what is expected in each.

1.2 Scope

1.2.1 This SOP primarily addresses instrument run log maintenance, maintenance manuals and other logs not addressed in other SOPs. Standards 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 is discussed in Laucks SOP LTL-1019.

1.3 Definition of Terms

1.3.1 Logbook - Any bound or unbound document that forms a record of activities and pertinent data regarding an activity including but not limited to maintenance logs, standards logs, reagent chemical logs, analysis logs including instrument outputs (computer generated or strip chart recordings), balance and temperature logs, or any other regularly maintained record of activity.

2. 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 logbook should be numbered and controlled according to procedures outlined in Laucks SOP LTL-1019. It is the analysts 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 ICPs, AAs, spectrophotometers, ion chromatographs, etc. have instrument maintenance manuals associated with the specific instrument.

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. 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.

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 GC, GC/MS, HPLC, GPC and other 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 current logbook forms (at the time of this writing) are located in Appendices I (GC/MS), II (GC and HPLC), and III (GPC). 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 GC, GC/MS, HPLC, GPC or other run, all of the pertinent information should be filled out for each injection.

3.3.4 The standards, samples, calibration checks, reference materials, QC samples, etc. should be listed in the order that they were analyzed.

3.3.5 Logbook information should be either completely filled out, or a logbook designed to incorporate all of the pertinent elements for that analysis so that all fields are filled in. Logbooks should contain all of the necessary information to track what analyses occurred, the processing order, and critical run parameters (such as what GC column was in use).

3.3.6 No empty space should be left between daily logbook entries. The end of the analytical sequence should be clearly marked and empty space on the page crossed out, the accepted practice being with a "Z" which covers the entire space being crossed out. This "Z" should be initialed and dated by the analyst making it.

3.3.7 The other type of run-log typically in use is the individual, loose-leaf instrument run-log printout. Where the instruments themselves don't produce such printouts, handwritten run-logs are produced by the analyst. These are the log types typically 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. 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.

3.4 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.

Appendix I

GC/MS Run Logs

Laucks T[illegible]g Labs, Inc.
GC/MS VOA [illegible]ument Run Log

IS /SS Amt: 1µl of _____ µg/ml
IS /SS Ref # :_____
Date : _____

Analyst: _____
Calibration Std Ref.: _____
Spike Sol'n Ref.:_____

624 8260 524 SIM CLP-Low CLP

#	FILE ID	SAMPLE ID	INJ. TIME	SAMPLE AMT	DF[1]	pH[2]	COMMENTS

[1] DF = 1 unless otherwise indicated.
[2] pH = 2 for waters unless otherwise noted.

Laucks Testing Laboratories

GC/MS Semivolatiles Injection Logbook

IS Std ID_____

CCV ID_____

DFTPP ID_____

Page: 5699

Date:_____

	File ID	Lab ID	Inj. Time	Sample Info	Dilution	Comments	Analys
1							
2							
3							
4							
5							
6							
7							
8							
9							
10							
11							
12							
13							
14							
15							
16							
17							
18							

Appendix II

GC/HPLC Run Logs

Laucks Testing Labs

GC VOA Instrument Log

Analyst : _____ Date : _____

Chromatography Ref. Page : _____ Calibration Reference : _____

File #	Sample ID	pH	Sample Vol/Wt	STD ID / AMT	SUR ID	Comments

PAGE :

LAUCKS TESTING LABORATORIES

INSTRUMENT LOGSHEET

Analyst : _____

Date : _____

Instrument ID : _____

Column 1: _____

Injection Volume : _____ ul

Column 2: _____

Calibration Standard Reference : _____

File ID	Sample ID	DF	Comments

LAUCKS TESTING LABORATORIES

HPLC INSTRUMENT LOGSHEET

Analyst : _____

Date : _____

Instrument ID : _____

Column : _____

Injection Volume : _____ ul

Solvent : _____

Calibration Standard Reference : _____

File ID	Sample ID	DF	Matrix	Comments

Appendix III

GPC Run Log

Laucks Testing Labs, Inc.
GPC Bench Sheet

Date	
Cal. Ref	
Analyst	
Case #	
SDG #	
Matrix	
Program	
Load Time	
Dump Time	
Collect Time	
Wash Time	

Operating Conditions:

N2 Pressure		
tank, high		psi
tank, low		psi
line		psi
Rinse Press.		psi
Sys. Press.		psi
Flow Rate		ml/min
Temp		deg F
Chart Speed		cm/min
Chart Full Scale		Volts
UV Detector		AUFS
Column ID		

Port	LTL Number	Client ID	Intermed Vol (ml)	Aliquot Clean (ml)	Volume Collect (ml)	Final Vol (ml)
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
21						
22						
23						

Comments:

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1008

Title: **QC Corrective Action**

Revision history:

Number	Date
5	6/22/96
4	3/3/96
3 (LTL-0008)	6/29/89
2 (LTL-0008)	5/19/87
1 (LTL-0008)	12/12/86

Written by: Harry Romberg, Quality Assurance Officer Date: 6-25-96

Approved by: Karen J Kotz, Laboratory Director Date: 6/25/96

UNCONTROLLED

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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.

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 **Reagent blank**: a measured volume of reagents used in a method.

2.1.5 **Method blank**: a reagent blank that undergoes a preparation (digestion, extraction, distillation, etc.) step prior to analysis.

2.1.6 **RPD**: Relative Percent Difference

2.1.7 **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.

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 data are affected.

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 reagent blank containing a target analyte greater than the method reporting limit.

3.1.1.9 A method blank containing interference or a target analyte at a concentration greater than or equal to the method reporting limit.

3.1.1.10 **Note:** 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.

3.1.1.11 A sample received, prepared or analyzed past holding time.

3.1.1.12 A sample depleted before all required analyses are completed.

3.1.1.13 An extract blown down to dryness, spilled or otherwise compromised.

3.1.1.14 Contaminated reagents and glassware.

3.1.1.15 Equipment malfunction or instrument failure, such as cold storage unit temperature outside acceptable ranges and the loss of data acquisition.

3.1.1.16 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; also maintains QC charts.

3.2.2.2 The analyst is also responsible for performing the following steps to correct the problem:

3.2.2.3 Examine all calculations for correctness

3.2.2.4 Examine bench sheets for correctness

3.2.2.5 Check instrumentation and operating conditions to preclude the possibility of malfunctions or operator error

3.2.2.6 Verify integrity of spiking solution, laboratory control sample, or calibration standard

3.2.2.7 Re-analyze the sample

3.2.2.8 Take other actions as noted in the specific analytical SOP.

3.2.2.9 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 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 all results that are suspect 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 which meet specifications, it may be determined that sufficient action has been taken. 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.

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, insure 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 been taken. The supervisor and QA will approve of such decisions.

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.

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 event the out-of-control event is due to matrix problems in the sample, and the system remained out of control, the data is flagged and supporting documentation is released to the client.

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.

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 affect reported data.

3.4.4 Audit Finding Reports are responded to by the assigned individual 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 allows data to be reported which is in control, the action is complete and only needs to be signed by the individual taking action and the individual initiating the action.

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 I

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_____96_____	☐	
MS/MSD	K_____96_____	☐	
SRM	R_____96_____	☐	
Blk Spk	S_____96_____	☐	
MS/Dup	M_____96_____	☐	
Duplicate	D_____96_____	☐	

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: _____
Date: _____

Distribution:
Original to QA
Copy to workorder / SDG file for all validatable packages and to first workorder on list for non-validatable data.

Appendix 4

Sample Receipt Form

Laucks Testing Laboratories, Inc.
SAMPLE RECEIPT LOG (1) CLP

P:\PM\Holly\docs\fax\ncmemo.doc 12/22/94

Initial once samples are checked in_____

DATE RECEIVED:_____
TIME RECEIVED:_____
CLIENT NAME:_____
SDG #_____
COC #_____

SAMPLE LOG-IN DATE:_____
WORKORDER #:_____
CLIENT PROJECT:_____
AIRBILL ATTACHED?:(#)_____
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:

Appendix 5

Example Logsheet (Cold Storage)

Cold Storage T[illegible]rature Log

Laucks Testing Laboratories, Inc.

Cold Storage ID #:

Location: Year: 1996 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												

REFERFRM DOC 5/29/96

Record Time and Temperature in the proper blocks and initial the entry each day of normal laboratory operation.

If refrigerator temperatures exceed 4°C±2°C or if freezer temperatures are warmer than -10°C, corrective action must be taken.

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.

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1009

Title: **Blind Spike Program**

Revision history:

Number	Date
LTL-0048 Rev1.1	05/18/92
2	06/21/96

Written by: Harry Romberg, QA Officer Date: 6-21-96

Approved by: Karen Kotz, Laboratory Director Date: 6/21/96

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1. Introduction and Scope

1.1 Description

1.1.1 This SOP provides a description of how blind spikes are generated, what types of analyses are monitored, how results are evaluated and how Laucks handles out of specification events.

1.1.2 Materials may be from a multitude of sources. The analyst will most often be aware that the sample is a blind spike but in no case should the analyst know the "true" value of the submitted sample. On 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 an EPA 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. ERA, APG or other sources of materials will be used where components are not present in WP, WS or other "official" PE samples. Acceptable results from programmatic samples, such as those for HAZWRAP, 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 analyzed semiannually (WP in approximately June and November, WS in approximately April and September). Supplementary PE samples for analytes not present in these samples (such as fuels or GC/MS semivolatiles) are generally obtained from APG, ERA or a similar vendor and are generally analyzed along with remedial samples (if any) resulting from WP failures (results being obtained approximately 3 months after submittal of the WPs). Other external PE samples from programs such as NFESC, HAZWRAP, 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.

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 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 (Pb, As, Se, Tl)
- Mercury
- GC Volatiles
- Gas/BTEX water & soil
- Diesel water & soil
- Petroleum Hydrocarbons (418.1) water & soil
- Pesticides
- GC/MS Volatiles
- GC/MS Semivolatiles

- PNAs
- Explosives
- 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.

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
4	1/30/98
3	6/21/96
2	1/08/96
1.1	6/15/90

Revised by: Harry Romberg, QA Officer Date: 1-30-98

Approved by: Karen J. Kotz, Laboratory Director Date: 1/30/98

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1. Introduction and Scope

1.1 Overview

1.1.1 This SOP describes the determination of Instrument Detection Limits (IDLs), Method Detection Limits (MDLs), Precision and Accuracy Studies, the setting of Reporting Limits and the determination and use of control limits. All are defined in the definitions section of this SOP.

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) Inorganic Statement of Work (SOW) and SW846.

1.1.3 Criteria for Precision and Accuracy (P&A) Studies are generally 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.

1.1.4 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.

1.1.5 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. Other MDLs may be estimated based on these studies.

1.1.6 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.

1.1.7 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 MDLs. This SOP is to be followed to validate a new method or to validate a change in a current method.

1.1.8 MDLs should be determined approximately annually for common procedures and as needed for procedures which may be performed on an infrequent basis.

1.1.9 PCB MDLs are to be performed for each PCB to be analyzed. At least one PCB MDL must be determined annually and all PCB MDL determinations must be performed within 3 years.

1.1.10 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 competency of the analyst.

1.2 Method Description

1.2.1 Detection Limits

1.2.1.1 For any metals method, 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 are generally 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.

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 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 often but not always defined in the method. 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 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 competency 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 most often defined in the reference method, although if the method performance has been demonstrated, analyst competency may be demonstrated in comparison to laboratory limits.

1.2.3 Control limits may be specified in a reference method 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.

1.2.4 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.

1.3 Definition of Terms

1.3.1 **Accuracy** - The degree of agreement of a measurement (of 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 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.

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 2 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** - 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.

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:

$$s = \sqrt{\frac{\sum (X_i - \overline{X})^2}{n-1}}$$

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.

3.1.2 Waste Disposal

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 QA is required.

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 they perform. In addition, each organic instrument analyst is responsible for verifying a valid annual MDL was performed on each instrument for each method they perform.

5.1.2 Each analyst is responsible for producing a one-time initial demonstration of precision and accuracy.

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, 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 Supervisor or Senior Analyst

5.2.1 Each area 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.

5.2.2 It is the responsibility of the area supervisor or senior analyst to insure that the analyst is performing the study 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.

5.3 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, 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. All studies will use the SAM client code QC_Officer in order to better track them at a later date.

6.2 Instrumental Detection Limits (IDLs)

6.2.1 It is rarely necessary to perform actual IDL studies except for metals analyses. For metals analyses, they are performed quarterly on each instrument. 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, GFAA, etc.)
- Analysts signature & date signed
- Supervisor or senior analyst review signature & date signed

6.2.6 Spectrophotometry

6.2.6.1 The 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.

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. In addition, the EPA/CLP method does 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.

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 MDL studies must be performed annually for each method for inorganic analysis and for each method/instrument combination that will be used for organic methods.

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 workorder ID must be obtained from QA. The data generated from the study is then referenced to that workorder 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).

- 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 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.

> **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.

6.3.8.1 Spiking levels which are determined to be less than 1x or greater than 10x the MDLs should in almost all circumstances be re-analyzed at a more appropriate spiking level.

6.3.8.2 Prepare reagent (blank) water that is as free of analyte as possible. Reagent or interference free water is defined as a water sample in which analyte and interference concentrations are not detected at the estimated method detection limit of each analyte of interest. Interferences are defined as systematic errors in the measured analytical signal of an established procedure caused by the presence of interfering species. The interference concentration is presupposed to be normally distributed in representative samples of a given matrix.

6.3.9 Preparation of Spiked Samples

6.3.9.1 The MDL is almost always determined in reagent water or clean sand. 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, 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:

$$s = \sqrt{\frac{\sum (X_i - \overline{X})^2}{n-1}}$$

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.

6.3.11 Methodology Exceptions/Specifics

6.3.11.1 Wet Chemistry

6.3.11.2 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.

6.3.11.2.1 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.

6.3.11.3 GC and Gas Chromatography/Mass Spectophotometry

6.3.11.3.1 The prime consideration in GC/MS determinations is the ability to make compound confirmation based on spectral identification criteria. For SIM methods this does not apply.

6.3.11.3.2 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 generally determined in one of four ways:

- Administrative decision
- Set equivalent to the Practical Quantitation Limit (PQL)
- Project Specific Requirements
- The low standard

6.4.2 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.

6.4.3 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. 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.

6.4.3.1 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. 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.

6.4.4 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.

6.4.5 On occasion, 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.

6.4.6 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 and in the ProjQC database. 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. 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. Any exception to the use of the CLP limits in these instances must also be noted in the ProjQC database and on any paperwork defining the details of the project.

6.5 Precision and Accuracy 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 will be required to 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.

6.5.5 All P&A studies must include the following information:

- 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 criteria or defer to a similar technical method with P&A criteria and use this P&A criteria as guidance in establishing performance criteria. In the case of organic SW846 methods, if the criteria 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. In most methods where criteria are defined, 4 replicates must meet method-specified criteria for the **laboratory** to be considered capable of adequate performance.

6.5.8 The individual **analyst** must be able to analyze four replicates and meet laboratory blank spike control limits to be considered competent to perform the applicable analysis. 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.

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 criteria.

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, 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 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 even though the

laboratory may be demonstrating adequate performance on the control material in any specific analytical run, it cannot demonstrate adequate performance for all samples in that run on a routine basis.

6.6.7 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.

6.6.8 The laboratory may be called upon to utilize control limits specified in a method or in a specific contract as designated in the LIMS ProjQC database or 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.

6.6.9 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.

7. Reports

7.1 Data Package Organization

7.1.1 All work, with the exception of control limit computations, is performed under laboratory workorder 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), ILM04.0.

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

Navy Installation Restoration Laboratory Quality Assurance Guide, Naval Facilities Engineering Service Center, February 1996

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 Information Sheet

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.

SAM MDL Name: _____

SAM Workorder Number: _____

Analysis:

Analyst: _____

Analysis Method: _____

Analysis Date: _____

Instrument ID: _____

Preparation:

Prepared by: _____

Preparation Method: _____

Preparation Date: _____

Cleanup Method(s): _____

Review:

Analyst's Signature: _____

Supervisor's Signature: _____

QA Approval: _____

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP No. **LTL-1012**

Previous SOP No. **LTL-0033**

Title: **Solvent QC Monitoring for Trace Residue Analysis**

Rev. 1

Laucks is in the process of re-numbering our SOPs. As an interim measure, this page serves as the cover page for those SOPs whose header information has not been updated. This page details the title, the SOP number that it is being controlled under, and the previous SOP number. The previous SOP cover sheet has been manually corrected to reflect the change but each page header will reflect the old numbering system. As SOPs are revised, the full header and cover page will be updated.

UNCONTROLLED

TO: Users of this SOP

After you have read this SOP you need to sign the operators statement which is attached to the master copy. See the QCO for access to this copy.

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP No. **LTL-1012**

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UNCONTROLLED

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-~~0033~~-~~[illegible]~~
1012

Title: Solvent QC Monitoring for Trace Residue Analysis

Submitted by: Harry Romberg
Harry Romberg

Date: 4-10-91

Reviewed by: Vicki Talksabout
Vicki Talksabout

Date: 4-11-91

Reviewed by: Mike Nelson
Mike Nelson

Date: 4/11/91

Approved by: Jim Owens
Jim Owens

Date: 4/15/91

TO: Users of this SOP

After you have read this SOP you need to sign the operators statement which is attached to the master copy. See the QCO for access to this copy.

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SECTION I - Introduction and Scope

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.

SECTION II - Equipment List

Glassware, reagents and equipment as delineated in the methods specific to the described task.

SECTION III - Safety Precautions

Typical precautions should be taken when handling any solvent. Some, such as methylene chloride are not flammable, but others, such as acetone and hexane are and should be treated with extreme caution. Long term health effects of solvent contact are 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.

SECTION IV - Operation procedures

Part 1. Initiation, Data Handling, and Record Maintenance

1.1 A new lot of any solvent must be sequestered by the supplier and the checking process initiated at least two 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.

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. Any larger amount of hexane or acetone may be ordered, if desired and if the solvent locker will accommodate it. No more than 4 months supply of methylene chloride will ever be ordered, as typical methylene chloride recommended shelf-life is 6 months. For methylene chloride, multiples of a 27 case pallet will be most conveniently ordered and delivered in shrink wrap plastic. Thus, the palette may be easily set aside until testing has been completed. Methylene chloride should be kept cool and in an low light area to inhibit breakdown.

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. 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.

1.4 The Extractions supervisor or designated representative initiates the checking process. When a bottle from a new, previously untested, lot of solvent is received, a Solvent Check Order form is filled out (Appendix I) designating the Manufacturer, lot number, solvent, tests to be performed and person initiating the testing. This form is given to the Sample Entry Clerk who creates a work order in SAM and gives it a work order number. One laboratory work order is established for EACH lot and type of solvent in order to very sharply distinguish between which are acceptable and which fail.

1.5 When testing has been completed, the lot will be officially designated as acceptable or failed by the QC Officer or Chief Chemist. This will be done by initialing the final report and contacting the Extractions supervisor. In fact, any lot will be considered acceptable which meets the criteria specified in Appendix II. As long as those criteria are met, the lot will be considered acceptable. 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.

1.6 The data and report files will be maintained by the QC Officer in a designated location specific for this purpose.

Part 2. **Solvent Analysis**

2.1 Methylene Chloride Acidity

2.1.1 .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.

2.1.2 Neutral ethanol - Add 25 mls. of denatured ethanol to an Erlenmeyer flask. Add 2 or more drops of phenolphthalien indicator solution (1 gm. phenolphthalien/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.

2.1.3 Add 25 mls. of the methylene chloride to be checked to the flask containi the 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.

2.1.4 Add 900 uL of the .01 N NaOH. Swirl to mix well.

2.1.5 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.

2.1.6 A "PASS" or "FAIL" is entered into the SAM report under the associated regular SAM test code, MECLAG. 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 QC Officer.

2.2 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.

2.3 In all cases, 500 mls. of the appropriate solvent is concentrated to 1 ml. in a Kuderna-Danish concentrator. No splitting of the concentrate occurs. Surrogates are not added.

2.4 Methylene Chloride - $MeCl_2$ is used for both ABN and pesticide/PCB analyses. A separate 500 ml. concentration is done for each analysis.

2.4.1 For the pesticide/PCB analysis, hexane is added and the solvent exchanged and concentrated down to 1.0 ml., which is submitted for analysis.

2.4.2 For ABN analysis nothing is added and the $MeCl_2$ concentrated directly down to 1 ml and submitted for analysis.

2.5 Acetone - Acetone is used for both ABN and pesticide/PCB analyses. A separate 500 ml. concentration is done for each analysis.

2.5.1 For the pesticide/PCB analysis, hexane is added and the solvent exchanged and concentrated down to 1.0 ml., which is submitted for analysis.

2.5.2 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.

2.6 Hexane - Hexane is used only in pesticide analysis. It will be concentrated 500 mls. to 1 ml. as stated and submitted for TCL pesticide analysis.

2.7 Acceptance criteria are compiled in Appendix III and are based on 500 mls of solvent concentrated to 1 ml. final volume. They are derived from EPA CLP criteria for acceptable blanks. The SAM report indicates the acceptance level, the level found and signifies whether the detected level (if any) passes (OK) or fails (FAIL).

APPENDIX I

Solvent Check Order Form

LAUCKS TESTING LABORATORIES

Solvent Check Order Form

Solvent:_____ Manufacturer:_____

Lot No:_____ Date:_____ Requested by:_____

SAM Number:_____

Tests to be performed:

☐	Methylene Chloride:	Acidity	(MECLAC)
		ABN QC	(MSQCCK)
		Pesticide/PCB QC	(PXQCCK)
☐	Acetone:	ABN QC	(MSQCCK)
		Pesticide/PCB QC	(PXQCCK)
☐	Hexane:	Pesticide/PCB QC	(PXQCCK)

APPENDIX II

Solvent Acceptance Criteria

Solvent Acceptance Criteria

Semivolatile Compounds	Total ng in 1 ml.
Phenol	10000
bis(2-Chloroethyl) ether	10000
2-Chlorophenol	10000
1,3-Dichlorobenzene	10000
1,4-Dichlorobenzene	10000
1,2-Dichlorobenzene	10000
2-Methylphenol	10000
2,2'oxybis(1-Chloropropane)	10000
4-Methylphenol	10000
N-Nitroso-di-n-propylamine	10000
Hexachloroethane	10000
Nitrobenzene	10000
Isophorone	10000
2-Nitrophenol	10000
2,4-Dimethylphenol	10000
bis(2-Chloroethoxy)methane	10000
2,4-Dichlorophenol	10000
1,2,4-Trichlorobenzene	10000
Naphthalene	10000
4-Chloroaniline	10000
Hexachlorobutadiene	10000
4-Chloro-3-methylphenol	10000
2-Methylnaphthalene	10000
Hexachlorocyclopentadiene	10000
2,4,6-Trichlorophenol	10000
2,4,5-Trichlorophenol	25000
2-Chloronaphthalene	10000
2-Nitroaniline	25000
Dimethylphthalate	50000
Acenaphthylene	10000
2,6-Dinitrotoluene	10000
3-Nitroaniline	25000
Acenaphthene	10000
2,4-Dinitrophenol	25000

Solvent Acceptance Criteria

Semivolatile Compounds	Total ng in 1 ml.
4-Nitrophenol	25000
Dibenzofuran	10000
2,4-Dinitrotoluene	10000
Diethylphthalate	50000
4-Chlorophenyl-phenyl ether	10000
Fluorene	10000
4-Nitroaniline	25000
4,6-Dinitro-2-methylphenol	25000
N-nitrosodiphenylamine	10000
4-Bromophenyl-phenylether	10000
Hexachlorobenzene	10000
Pentachlorophenol	25000
Phenanthrene	10000
Anthracene	10000
Carbazole	10000
Di-n-butylphthalate	50000
Fluoranthene	10000
Pyrene	10000
Butylbenzylphthalate	50000
3,3'-Dichlorobenzidine	10000
Benzo(a)anthracene	10000
Chrysene	10000
bis(2-Ethylhexyl)phthalate	50000
Di-n-Octylphthalate	50000
Benzo(b)fluoranthene	10000
Benzo(k)fluoranthene	10000
Benzo(a)pyrene	10000
Indendo(1,2,3-cd)pyrene	10000
Dibenz(a,h)anthracene	10000
Benzo(g,h,i)perylene	10000

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 and Reference Materials**

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UNCONTROLLED

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1. Introduction and Scope

1.1 Method Description

1.1.1 This SOP is intended to describe the way in which standards and reference materials are tracked, prepared, stored and maintained at Laucks, from the time of receipt of the neat or stock materials, solutions or their preparation to the point of use of the working standard. 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.

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.

2. Equipment Lists and Standards

2.1 Equipment

2.1.1 Equipment and reagents necessary for the preparation of any specific solution.

2.2 Standards

2.2.1 Standards as specified in each analytical SOP.

2.2.2 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 3/90 CLP Organic statement of work.

2.3 Standards Logbooks

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.

4. Operation Procedures

4.1 Preparation of Organics and Inorganics Materials

4.1.1 General consideration in standard preparations include:

4.1.1.1 Determine volumes and aliquots required using the concentration calculations in Appendix 1.

4.1.1.2 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.

4.1.1.3 Be sure to use a solvent volume sufficient to dissolve all analytes.

4.1.1.4 The solvent used should be miscible with water when being used for sample spiking purposes. Most standards used in the extractions laboratory are prepared with methanol.

4.1.2 Proper Syringe/Pipette Technique

4.1.2.1 Choose an appropriate size syringe so that the measured volume is at least 2/3 of the total volume of the measurement device.

4.1.2.2 When selecting a pipette, choose volumetric pipettes only for the exact amount to be measured.

4.1.2.3 Always rinse a syringe (organics) at least ten times with the appropriate solvent in between measurements, and wipe the syringe with a Kim-wipe.

4.1.2.4 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.

4.1.2.5 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.

4.1.3 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.

4.1.3.1 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.

4.1.3.2 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.

4.1.3.3 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 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.

4.1.3.4 For volatiles, the flask is inverted and gently mixed three times after diluting to the mark.

4.2 Traceability Documentation for Organics and Inorganics Materials

4.2.1 All organic neat standard materials are logged into the NEATS database, as described in 4.2.5, when they arrive in the lab. No neat organic material should be used before it has been logged into the database. Inorganic stock materials are logged directly into the appropriate standards logbook. Examples of some NEATS database screens are provided in Appendix 3.

4.2.2 All standard, spike, or surrogate mixes which are diluted solutions, whether organic or inorganic in nature, are not logged into the database but are logged directly into the appropriate standards logbook.

4.2.3 The current controlled logbooks are identified in each area as follows:

- GC/MS Volatiles - MV# (used for standards made from neat materials, single analyte concentrates, or supplier provided standard mixes)
- GC/MS Semivolatiles - MS#
- Metals - ME#
- GC Pesticides - PX#
- GC Volatiles - VOA#
- GC & HPLC PNAs - BA#
- other GC & HPLC analyses - MA#
- Organic Extractions misc - EX#
- Technicon & Lachat Analyzers - TE#
- IR Oil and Grease - IN#

- Ion Chromatography - IC#
- TOC/TOX - OC#

NOTE 1: # in the above table indicates a sequential number, beginning with 1, with each subsequent controlled book with that analysis code having the next higher integral value.

NOTE 2: This logbook number is for tracking standards only. The logbooks also will have a QA logbook number used for controlling logbooks which is independent of the standards tracking process.

4.2.4 All purchased stocks and subsequent standard preparations must be recorded in the appropriate database or log-book.

4.2.5 Upon receipt, each purchased neat material, stock, intermediate or working solution is entered into either the database (if an organic neat material) or a standards log-book and assigned a unique LAUCKS identification number. The information entered in the database or standards logbook must include:

- Analyte(s) name and vendor product ID (vendor ID must be given to unequivocally identify exactly what was used).
- supplier name
- supplier lot number
- concentration and/or purity
- expiration date (either vendor supplied, the analytical SOP or determined from the shelf life table in Appendix 2, in order of preference)

NOTE: In the case of the metals solutions which are supplied without an expiration date, the date opened and corresponding expiration date will be added when the standard is opened based on, in order of preference, the analytical SOP or Shelf Life table in Appendix 2.

4.2.6 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.

4.2.7 Every prepared stock, intermediate or working standard solution is entered into the standard log-book and assigned a unique LAUCKS ID number. The logbook entry must include the items detailed in section 4.2.9. Each material must be labeled with LAUCKS ID number, preparation date, expiration date and preparer's initials. Other items to be included on the label are listed in section 4.3.1. Examples of typical standards logbook entries are provided in Appendix 4.

4.2.8 An example of the solution nomenclature used is a working ABN standard prepared on 11/13/91. The solution number assigned was MS 2-77-2. This label represents the following:

- MS - solution was made and used as a semivolatile mass spec standard
- 2- solution was logged into standard book #2
- 77- page number on which solution has been recorded
- 2- this denotes the second entry on page 77

4.2.9 All discrete measurements made during a standard preparation must be recorded in the log book, specifically, weights aliquots and final volumes.

Other pertinent data to be entered in the log book are as follows:

- Standard Name
- Parent material and concentration/purity
- Solvent/Diluent standard is prepared in
- Type of standard being prepared (i.e. inter-mediate, spike, working, calibration)
- Final concentration
- Date prepared/opened
- Expiration dates
- Analysts initials

4.2.10 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. See Appendix 5 for examples of typical bench sheets with standards references.

4.3 Storage of Standards and Reference Materials

4.3.1 Always completely label solution with the following information:

- LAUCKS ID number
- Standard name
- Concentration
- Solvent/Diluent
- Technician's initials
- Date of preparation
- Expiration Date

4.3.2 Organic Standards and References Materials

4.3.2.1 Store in vial or bottle which minimizes head space.

4.3.2.2 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.

4.3.2.3 Volatile Standards and Reference Materials

4.3.2.3.1 All standards solutions should be stored in the VOA freezer at -10°C to -20°C.

4.3.2.3.2 Most volatile standards are stored in the original ampules until used.

4.3.2.3.3 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.

4.3.2.4 Other Volatile Standard Solutions

4.3.2.4.1 Some standards need to be prepared in the lab. Stock solutions are diluted using high purity MeOH.

4.3.2.4.2 To insure stability, standard solutions should be sealed in amber glass ampules

4.3.2.4.3 Rinse unsealed ampules with clean MeOH and place in oven to dry.

4.3.2.4.4 Cover ends of ampules with foil.

4.3.2.4.5 Dilute stock solution in high purity MeOH in a volumetric flask.

4.3.2.4.6 Mix gently.

4.3.2.4.7 Partially fill ampules with solution and recap with foil.

4.3.2.4.8 Use CO_2 to cool ampules until crystals form on sides.

4.3.2.4.9 Heat end of ampule with acetylene flame until glass begins to soften.

4.3.2.4.10 Gently pull end until seal is formed.

4.3.2.4.11 Label ampules and store in freezer.

4.3.2.4.12 Record the information in the Mass Spec VOA Standards Log Book (MV).

4.3.2.4.13 When standard solutions are used they should be transferred to Mininert cap vials with Teflon lined septa. The vials are stored in the VOA freezer until discarded.

4.3.2.5 Semivolatile Standards and Reference Materials

4.3.2.5.1 All standards solutions should be stored at a maximum temperature of 4 degrees C (± 2 degrees). Refer to the analytical SOPs for details as some analytes may drop out of solution if at cooler temperatures.

4.3.3 Inorganic Standards and Reference Materials

4.3.3.1 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 standards are kept at 4°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.

4.4 Shelf Life

4.4.1 Expiration

4.4.1.1 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, 01/96, the material expires after 1/31/96. This guidance was obtained from various vendors.

4.4.1.2 All parent expiration dates MUST be entered into the standard log books and the expiration date for all resulting child materials must also be entered into the logbook and placed on the material label.

4.4.1.3 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 05/20/95 in 01/95, instead of having a six month shelf life (07/95) the solution will expire, 05/20/95, the same date as the parent.

4.4.1.4 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.

4.4.1.5 If a standard is past its expiration date it may be used for qualitative purposes only. The standards logbook must be edited to reflect this status and 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".

5. Standard Verification

5.1 Criteria

5.1.1 Standards are to have their concentrations verified before use whenever possible. The QC'ing of the standard is to be recorded in the applicable column in the standards logbook unless they are validated in the individual analytical run (such as confirmation by another standard from an independent source). Criteria for standards acceptability are in many cases defined in individual SOPs. In instances where they are not so defined, acceptability criteria are:

- 80% - 120% for organics
- 90% - 110% for inorganics

Appendix 1

Example Calculations

1. Concentration Calculations from Neat Materials

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) $$0.2500gm * \frac{1000mg}{1.0g} = 250mg$$

A.1) Adjust the 250 mg for purity,

i.e. if purity = 90%, 250 mg x 0.9 = 225 mg

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)

2. Intermediate and Working Standards (Standard Dilution)

$$(FC)(FV) \times 1000 = (AV)(PC)$$

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

If the % purity is ≥ 97%, it is considered 100% pure 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]	6 Months *[2]
Prepared Stock Solution	12 Months	12 Months	6 Months
Intermediate Solution	3 Months	N/A	6 Months
Working Solution	2 weeks *[3]	6 Months	3 Months
Purchased Working Solution		6 Months	3 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.

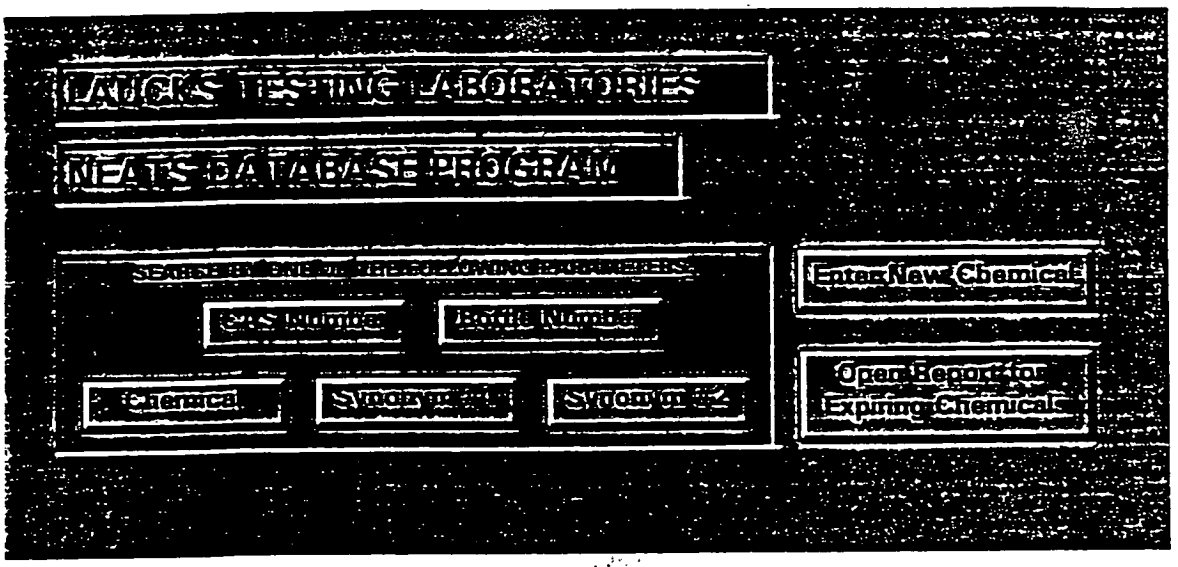
NOTE: THIS IS A GENERAL PROTOCOL. WHERE POSSIBLE, VERIFY THE INTEGRITY OF THE WORKING STANDARD SOLUTION BY MEETING THE ACCEPTANCE CRITERIA SPECIFIED IN THE ANALYTICAL SOP FROM THE KNOWN TRUE VALUE WHEN ANALYZED AGAINST AN INDEPENDENT LABORATORY CONTROL STANDARD OR A CALIBRATION CURVE.

A. Volatiles:

Method	Expiration Date
SW 846 8240 B	Stock Standards: 6 months; gases weekly if unstable, or 6 months if prepared in nitrogen. Calibration standards prepared daily
CLP OLM01.9	Stock Standards: 6 months or sooner. Stock gas standards: 2 months or sooner Secondary dilution standards: 6 months or sooner (gases & reactive compounds: monthly or sooner) Calibration standards: weekly or sooner. IS, surrogate & matrix spike: fresh spiking solution weekly or sooner. Aqueous standards: 24 hours at 4°C or 1 hour at room temperature; 12 hours if stored on autosampler.
CLP OLM0 2.0-03.1	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. 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.
10/92 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 every 3 months or sooner Surrogates: prepare fresh surrogate solution every 6 months or sooner
SW 846 8260A	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

Neats Database Screens


LAUCKS TESTING LABORATORIES
NEATS DATABASE PROGRAM
Enter New Chemicals
Open Report for Expiring Chemicals
Chemical

NEATS DATA ENTRY

Chemical:	n-nitroso-diphenylamine	CAS #:	[illegible]
Synonym #1:		Purity (%):	98.3
Synonym #2:		Lot #:	153-150B
Bottle #:	[illegible]	Source:	ChemService
Old Bottle #:		Catalog #:	O-374
Bottles:	2	Size (g):	2
Received:	4/3/96		
Expires:	10/1/2000		
Notes:	For GC/MS SVOA MS/MSD		

Previous | Next | New | Exit

Appendix 4

Logbook Examples

LAUCKS TESTING LABORATORIES, INC.
STANDARDS LOGBOOK

#	ANALYTE	ID or STOCK No.	STOCK CONC.	VOL/WT TAKEN	FINAL VOL.	CONC.	SOLVENT	PREP. DATE	INIT.	EXP. DATE	QC'D (Initials)
1	SW846 high std	ME3-96-01 10/19/96	LTL-CAL-1	10 mls	100 mls		HCL 5% HNO3 1% H2O	~~4/3/96~~ 4/2/96	KP	7/10/96	KP
		ME4-45-01 4/1/97	LTL-CAL-2	1 ml							
		ME4-~~32~~-02 3/14/97	LTL-CAL-3								
		ME3-96-05 7/10/96	1000 ppm Mo 7/10/96								
		ME3-96-09 7/10/96	1000ppm Pb 7/10/96	4 mls							
		ME3-97-08 11/17/96	1000ppm Cu 11/17/96								
		ME3-96-12	1000ppm Zn								
2	ABN SPE ICP/MS ICV3	ME4-30-01	ICP/MS KV-1	500 ul	5 mL		10% HNO3 H2O	4/3/96	ABN		
3	AS	ME3-81-9 LLP 5/96	1000ppm	100 µL	3-11-96 ~~10 mL~~ 100 mL	1ppm	5% HNO3	4-3-96	tm	5-96	
4	ICP/MS STD1	ME4-30-02	ICP/MS	500 µl	5 mL		5% HCL H2O	4/3/96 ABN	ABN		
5	STD2		Starch Std	250							
6	STD3			500							

LAUCKS TESTING LABORATORIES, INC.
STANDARDS LOGBOOK

#	ANALYTE	ID or STOCK No.	STOCK CONC.	VOL/WT TAKEN	FINAL VOL.	CONC.	SOLVENT	PREP. DATE	INIT.	EXP. DATE	QCD (Initials)
HM4-03-04 con't 4	2,4,6 - TNT	M-8330-11	1000 PPM	10 ml	50 ml	200 ppb	DI H2O	12/20/95	MH	04/01/96	
⊥	HMX	M-8330-4	⊥	⊥	⊥	⊥	⊥	⊥	⊥	⊥	
01	1,3,5 - TNB	M-8330-12	1000 PPM	10 ml	50 ml	200 PPB	Fluid TCLP #1	12/20/95	MH	04/01/96	
	2,4 - DNT	M-8330-2									
	RDX	M-8330-5									
	2,4,6 - TNT	M-8330-11									
⊥	HMX	M-8330-4					⊥				
02	1,3,5 - TNB	M-8330-12					TCLP #2 Fluid				
	2,4 - DNT	M-8330-2									
	RDX	M-8330-5									
	2,4,6 - TNT	M-8330-11									
⊥	HMX	M-8330-4	⊥	⊥	⊥	⊥	⊥	⊥	⊥	⊥	

LAUCKS TESTING LABORATORIES, INC.
STANDARDS LOGBOOK

#	ANALYTE	ID or STOCK No.	STOCK CONC.	VOL/WT TAKEN	FINAL VOL.	CONC.	SOLVENT	PREP. DATE	INIT.	EXP. DATE	QC'D (Initials)
1	IC ICV Std. Cl	ERA 3112	—	—	250 ml	129 mg/L	—	logged in 3/18/96	SK	Opened: 4/9/96 Exp: 4/19/97	
	F		—	—		6.47 mg/L	—				
	NO_3		—	—		8.77 mg/L	—				
	SO_4		—	—		11.9 mg/L	—				
2	Cal. Std. F	IC4.84.4	1000 ppb	500 µl	100 ml	5 ppb	H_2O	3/20/96	SK	4/3/96	
	Cl	.5	1000 ppb	1000 µl		10 ppb					
	NO_2	.7	100 ppb	2000 µl		2 ppb					
	NO_3	.3	1000 ppb	500 µl		5 ppb ~~10 ppb~~ 4/2/96					
	SO_4	.6	1000 ppb	2000 µl		20 ppb					
3	NO_2 ICV	IC4.78.7	100 ppb	1000 µl	100 ml	1 ppb	H_2O	3/20/96	SK	3/27/96	
4	NO_2 ICV	IC4.78.7	100 ppb	1000 µl	100 ml	1 ppb	H_2O	3/25/96	SK	4/1/96	

Appendix 5

Bench Sheet Examples

As, Tl

AutoSampler Report Table: SW846 Wed 04-10-96 09:39:45 AM page 1

Table Name: SW846 Autosampler Type: TYPE TJA
Sample Positions: 257/300 QC Positions: 11/19 # Sets: 1
Rinse Station location is rack -1, pos. -1.

--- Racks ---

Rack #	Type	Usage	#Pos Left	Analyses/Pos
1	Aux. (L) Rack	STD/QC/BLANK	11	10
2	Sample (13mm)	Samples	32	1
3	Sample (13mm)	Samples	75	1
4	Sample (13mm)	Samples	75	1
5	Sample (13mm)	Samples	75	1

--- Sample Sets ---

Set#	Type	Prepare?	Description	Method	#Pos	Rack#	StartPo
1	,Y	No	UMC09,UMC11 RE-AS,TL	UMASOIL	43	2	1

Report As, Tl
KP 4/10/96

--- Preparation Info ---

Set#	Uptake	Uptake#2	Final	Dil.Factor

No Samples Prepared.

Rack #1

Pos	Row	Col	Sample Name	Set #	#Used	Type
1	1	1	ICV1 ME4-48-01	-NA-	1	QC Standard
2	1	2	STD4 ME4-48-01	-NA-	1	Standard
3	1	3	STD3 ME4-48-01 5ml/10ml	-NA-	1	Standard
4	1	4	STD2 ME4-48-01 1ml/10ml	-NA-	1	Standard
5	1	5	STD1 ME4-45-02	-NA-	1	Standard
6	1	6	STD0	-NA-	1	Standard
7	1	7	Blank	-NA-	7	Blank
8	1	8	CCV ME4-51-01	-NA-	6	QC Standard

(9...19 Not Used)

Rack #2

Pos	Row	Col	Sample Name	Set #	#Used	Type
1	1	1	CRII1 ME4-45-02	1	-NA-	Sample
2	1	2	ICSABI1 ME4-52-05	1	-NA-	Sample
3	1	3	PBS1	1	-NA-	Sample
4	1	4	LCSS1	1	-NA-	Sample
5	1	5	03040-01	1	-NA-	Sample
6	1	6	03040-01D	1	-NA-	Sample
7	1	7	03040-01S	1	-NA-	Sample
8	1	8	03040-01L	1	-NA-	Sample
9	1	9	03040-02	1	-NA-	Sample
10	1	10	03040-03	1	-NA-	Sample
11	1	11	03040-01 5X	1	-NA-	Sample
12	1	12	03040-01D 5X	1	-NA-	Sample
13	1	13	03040-01S 5X	1	-NA-	Sample

LAUCKS TESTING LABORATORIES
ABN GC/MS OPERATIONS LOG

CASE # _____

PAGE 4883-A

3/18/96 IS = MS334-1 DFTPP = MS334-4

RUN #	FILE NAME	CLIENT NAME	CLIENT SAMPLE#	LAB I.D. #	INJECTION INFO	IS A RT	IS A RESPONSE	IS B RT	IS B RESPONSE	IS C RT	IS C RESPONSE
1	MH7501										
2	HC18A	—	SSTD050	MS4-9-1	25.% ABN	11.24	55075	14.99	159300	20.44	104485
3	HC18B	—	SSTD200	MS3-89-1	100.% w/ phenol	11.22	59100	14.98	169403	20.42	100469
4	HC180	Kalama	B031396MSWLA	9603368-BLK	1000→1	11.23	58139	14.98	166207	20.43	99304
5	HC181	Kalama	S031396MSWLA	—SPK	1000→1	11.24	55723	14.99	155289	20.44	98643
6	HC182	Olympia	—	9603206-01DL	1:4 1000→1	11.24	52364	14.98	158592	20.44	102071
7	HC183	Kalama	—	9603368-01	1:2 100→1	11.24	56997	14.98	168568	20.46	103091
8	HC184	↓	—	-01DL	1:4 100→1	11.24	58326	2,3,4,6 N 15.00	173480	20.45	106213

3/19/96

LAUCKS TESTING LABORATORIES INC.
Seattle. Washington

SOP #:LTL-1017

Title: Internal Audit Procedures

Revision history:

Number	Date
000	5/13/93
001	3/3/96

Written by: Harry Romberg Date: 3/3/96
Harry Romberg

Approved by: Karen T Kotz Date: 3/3/96
Karen T. Kotz. Laboratory Director

UNCONTROLLED

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1. Introduction and Scope

1.1. Method Description

1.1.1. The purpose of this procedure is to provide instructions for planning, performing and reporting QA/QC audits within the laboratory.

1.1.2. This method is restricted to use by, or under the supervision of personnel experienced in the technique described.

1.2. Discussion

An Audit of the facility is performed for the following reasons:

1.2.1 To determine that contractual and regulatory obligations are fulfilled.

1.2.2 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.

1.2.3 To establish that quality assurance objectives are met, including holding times, use of approved analytical methods, and stated objectives for precision and accuracy.

1.2.4 To serve as a management tool to evaluate appropriateness of quality assurance policies.

1.2.5 To identify potential or actual deficiencies for the purposes of evaluating compliance with requirements and providing the means for correction.

1.2.6 To determine that records are prepared and maintained as required.

1.3 Documentation and Frequency

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

1.4.3 This section defines terms and acronyms as they are used in this SOP.

1.4.4 SOP: Standard Operating Procedure

1.4.5 QA: Quality Assurance

1.4.6 QC: Quality Control

1.4.7 Audit: A planned and documented activity performed to determine by investigation, examination, or evaluation of objective evidence the adequacy of and compliance with established procedure, instruction, and other applicable documents and the effectiveness of implementation. An audit should not be confused with surveillance or inspection activities performed for the sole purpose of process control or product acceptance.

1.4.8 Auditor: Any individual: who performs or assists in the performance of any part of an audit, including technical specialists.

1.4.9 Lead Auditor: An individual who is qualified to organize and direct an audit, report audit findings, and evaluate proposed corrective actions.

1.4.10 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** discrepancy in the audit report.

1.4.11 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.

1.4.12 Discrepancy: Departure from approved procedures, program requirement, 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.

1.4.13 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.

2. Responsibilities

2.1 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.

2.1.1 QA Manager is 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

2.1.2 If an audit team is used. the following responsibilities fall upon the Audit Team Leader. If an audit team is not used. the following responsibilities fall to 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

2.1.3 Management of audited departments is responsible for the following:

- 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.
- Verifying initial implementation of corrective action for deficiencies in their areas, if applicable.

2.2 Audits and reports are to be performed by personnel in the laboratory who have demonstrated the ability to evaluate processes in the laboratory with emphasis on Quality Control and Quality Assurance.

2.3 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.

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. Calibration and Quality Control

Not applicable.

5. Operation procedures

5.1 General

5.1.1 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.

5.1.2 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 selection of an audit team.

5.1.3 Audit team members shall have received appropriate indoctrination and training for auditing.

5.2 Audit Planning

5.2.1 The QA Manager. or designee shall develop an audit plan which shall be the basis for the audit. The audit plan is documented on Audit Plan Form (Se Appendix I).

5.2.2 The QA Manager shall develop an audit checklist appropriate to the activity or area being audited. The checklist should contain auditable requirements extracted from the QA Manual,

applicable SOP's or guidance documents. such as EPA SW846. Checklists are designed for each Department by the QA Manager and can be accessed by the QA Department.

5.2.3 The QA Manager shall ensure that the checklist provides an adequate means for indicating whether the question is satisfactorily answered.

5.2.4 Audits are scheduled in a manner to provide coverage and coordination with ongoing QA program activities.

5.2.5 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.

5.2.6 The QA Manager notifies the audited department. in writing. prior to the audit to provide the subject and scope of the audit. audit schedule. and audit team members. if applicable.

5.3 Audit Performance

5.3.1 The QA Manager and (when required) the appointed audit team members shall proceed through the audit checklist recording evidence of compliance. discrepancies. or recommendations.

5.3.2 During the audit. the QA Manager or appointed team member 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.

5.3.4 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.

5.3.5 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.

5.4 Audit Report

5.4.1 The QA Manager or his designee shall prepare an audit report which should address the following:

5.4.1.1 Date and location (Laucks-department) of the audit.

5.4.1.2 Purpose and scope of the audit.

5.4.1.3 Audit team members (when applicable) and the people contacted during the audit.

5.4.1.4 Description of items. including the rank. type and detail of the audit finding requiring corrective action. The description of the items must be in sufficient detail to enable investigation. evaluation. and correction of the finding. (See Appendix II - Audit Finding Report Form) The report may also include the area affected (See Table in Appendix III) and Finding Type (See Table in Appendix IV)

5.4.1.5 Due date for completion of corrective action plans.

5.4.2 The QA Manager shall issue the audit report to the appropriate levels of Laucks management within four following the audit. This report shall include a copy of each finding. deficiency and/or recommendation.

5.5 Audit Closure and Follow-Up

5.5.1 The appropriate Laucks Management (departmental supervisors, laboratory director hall investigate the reported finding. deficiency or recommendation and do the following:

5.5.1.1 Determine the actions required to correct the discrepancy.

5.5.1.2 Evaluate each discrepancy to determine the root cause of the problem and any generic implications.

5.5.1.3 Determine the corrective action required to correct the discrepancy and to prevent recurrence.

5.5.1.4 Document corrective action and indicate corrective action commitment date.

5.5.1.5 Sign. date. and return the completed form to the QA Manager within the assigned time frame given in the audit report.

5.5.2 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.

5.5.3 The QA Manager shall verify the corrective action, as stated in the response, and make sure it has been implemented and accomplished as scheduled.

5.5.4 An interim status report of corrective action completion may be issued

5.5.5 After verification of corrective action. the QA Manager shall issue a report stating that all corrective action has been completed and the audit is closed.

5.5.6 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.

5.6 Records

The QA Manager shall ensure that the following audit documentation is maintained on file:

5.6.1 Completed audit checklist.

5.6.2 Audit Report (includes findings. deficiencies and recommendations).

5.6.3 Corrective Action (response to discrepancies).

5.6.4 Records pertaining to the completion of corrective action.

5.7 Audit Discrepancy Tracking

5.7.1 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

5.7.2 Explanations of Categories Listed Above

5.7.2.1 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.

5.7.2.2 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, HTVRs, and forms), records being stored in work areas for longer than 6 months, improper handling of controlled procedures.

5.7.2.3 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.

5.7.2.4 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 the applicable corrective action from.

5.7.2.5 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.

5.7.2.6 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 use of standards or solvents, lack of certification documentation, etc.

5.7.2.7 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.

5.7.2.8 Safety and reagent/chemical labeling findings involve any deviation from approved safety and waste procedures and the chemical hygiene plan.

5.7.2.9 Training and training records findings involve lack of training records, and personnel performing analysis without appropriate qualification documentation.

5.7.2.10 Good Laboratory Practice findings involve significant figures, temperature monitoring, calibration techniques and other associated activities involved with safe and accurate laboratory practices.

6.1 References

Laucks Quality Assurance Plan

Applicable SOPs

Audit Database Tables

Appendix I

Audit Plan Form

LAUCKS Testing Laboratories

Audit Plan

Area to be Audited: _____

Lead Auditor: _____

Audit Team Members (if applicable): _____
_____

Date of Audit: _____

Type of Audit: _____

Checklist(s) to be Used: _____
_____
_____
_____

Individuals Contacted During the Audit: _____
_____
_____
_____

Audit Debrief Date: _____

Report Issued Date: _____

Signature of Lead Auditor: _____

Signature(s) of Team Members: _____
_____
_____

Appendix II

Audit Finding Report Form

Audit Finding Report

Audit Number: Example	Finding Number 1
Facility:	Audit Date:
Auditing Body:	Audit Type:
Lead Auditor:	Affected Area: GC-Semivolatiles

Related Findings:

Finding Rank: Minor

Repeat Finding?: No

Finding:

Corrective Action Response:

Opened By:	Date Opened:
Response By:	Response Date:
Corrective Action By:	Scheduled Completion Date:
Verified By:	Date Verified:

Appendix III

Area Names

Department

Depart	Department	DepartmentDescription	Sup ID #
ARCH	Archive	Archive of Documents in QA	0006
BP	Bottle Prep	Bottle Prep	0008
DM	Data Management	Data Management and Administrati	0008
EXT	Extractions	Extractions	0027
GCEF	GC-Extractable Fuels	Extractable Fuels by GC/FID	0038
GCS	GC-Semivolatiles	GC-Semivolatiles	0048
GCV	GC-Volatiles	GC-Volatiles	0038
MSS	GC/MS-Semivolatiles	GCMS-Semivolatile	0048
MSB	GC/MS-Semivolatiles & Volatile	GC/MS-Semivolatiles and Volatiles	
MSV	GC/MS-Volatiles	GCMS-Volatile	0038
SAF	Health and Safety	Health and Safety	0006
HPL	HPLC	HPLC	0038
IN	Inorganics	Metals and Wet Chemistry Office	0053
MIS	LIMS and MIS	LIMs and MIS	0070
MET	Metals	Metals and Metals Prep	0067
MTI	Metals Instrumentation	Metals Instrumentation	0067
MTP	Metals Preparation	Metals Preparation	0067
PM	Project Management	Project Management	0008
QA	Quality Assurance	Quality Assurance	0006
SM	Sales and Marketing	Sales Department	
SC	Sample Control	Sample Control	0008
SP	Special Chemistry	Special Chemistry	0053
WC	Wet Chemistry	Wet Chemistry	0053
YAK	Yakima Office	Yakima Office	0072

Appendix IV

Finding Type

FINDING DEFINITION

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
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
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 GLPitems
MD1	Methods - No SOP/cribsheet available at time of audit
MD2	Methods - SOP/Cribsheet in use not current controlled version
MD3	Method- controlled SOP/Cribsheet 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 or handwritten SOPs in use
PE1	Performance Evaluation Samples - Results are outside warning limits, check for error
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	QAPlan - No QAP Available
QP2	QAPlan - Outdated And Needs Revision
QP3	QAPlan - Has Major Discrepancies With SOPs or practices of the day
RC1	Records Control - Logbooks Not Controlled
RC2	Records Control - Filing not maintained per SOP
RC3	Records Control - No SOPs to desribe 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
SF1	Safety - No SOP
SF2	Safety - Not Adhering to SOP or Chemical Hygiene Plan
SF3	Safety - Not Adhering to Good Lab Safety Practices
ST1	Stds/Reagents - No SOPs for preparation
ST2	Stds/Rgnts - Prep record inadequ./or not traceable
ST3	Stds/Reagents - Expiration Date Misused
ST4	Stds/Reagents - Not Labeled Properly in the laboratory
SW1	Software - Not Verified and Documented
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
TR1	Training - No Formal Program or Documentation
TR2	Training - Incomplete Forms (eg Proficiency, Hrs)
TR3	Training - Not Maintained Consistently

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	06/17/96

Written by: Harry Romberg, Quality Assurance Officer Date: 6-17-96

Approved by: Karen Kotz, Laboratory Director Date: 6/20/96

UNCONTROLLED

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1. Introduction and Scope

1.1 Description

1.1.1 This SOP is intended to provide an overview and general organization of data review practices employed for validatable packages. The actual data review processes and check lists specific to those types of analyses are covered in specific SOPs. A schematic diagram of the general review process is provided in Appendix I.

1.1.2 Validatable packages are often similar to the Contract Laboratory Program (CLP) presentation, although the actual analyses themselves and the applicable quality control (QC) may be from SW 846 or other references. If such is the case, the CLP format would be modified to meet the requirements of the referenced methodology. However, the overall review process remains the same.

1.1.3 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.

1.1.4 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 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. Responsibilities

3.1 Analyst

3.1.1 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 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 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.

3.2 Peer or Secondary Review

3.2.1 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. 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

3.3 Supervisor

3.3.1 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 but must include:

- Checking for reasonableness and sensibility of the reported 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

3.4 QA Review

3.4.1 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. 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
- Checking for deviations from Method or project QC requirements
- Checking for compliance with SOPs (periodically)

3.5 Project Manager

3.5.1 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
- 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.

3.6 Management

3.6.1 Senior management reviews case narratives and other components of data packages, should they find cause. They are the parties responsible for approving the release (signing) of reports.

4. Operation Procedures

4.1 General

4.1.1 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.

4.1.2 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.

4.2 Analyst

4.2.1 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.

4.2.2 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.

4.2.3 When completed with the data package, the analyst will pass all of the associated materials along to the second reviewer.

4.3 Peer or Secondary Review

4.3.1 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.

4.3.2 The secondary reviewer will pass the data and checklist along to the supervisor

4.4 Supervisor

4.4.1 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.

4.5 Reporting

4.5.1 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.

4.6 Quality Assurance

4.6.1 QA performs cursory reviews of most narrative 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.

4.6.2 QA 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.

4.7 Project Management

4.7.1 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.

4.8 Management

4.8.1 Management will review and release (sign) narratives.

5. Reports

5.1 Data Review and Signatures

5.1.1 Data review forms are provided in individual data review SOPs.

5.1.2 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.

5.1.3 Management signatures appear on all final reports.

6. References

Navy Installation Restoration Laboratory Quality Assurance Guide, Naval Facilities Engineering Service Center, February 1996

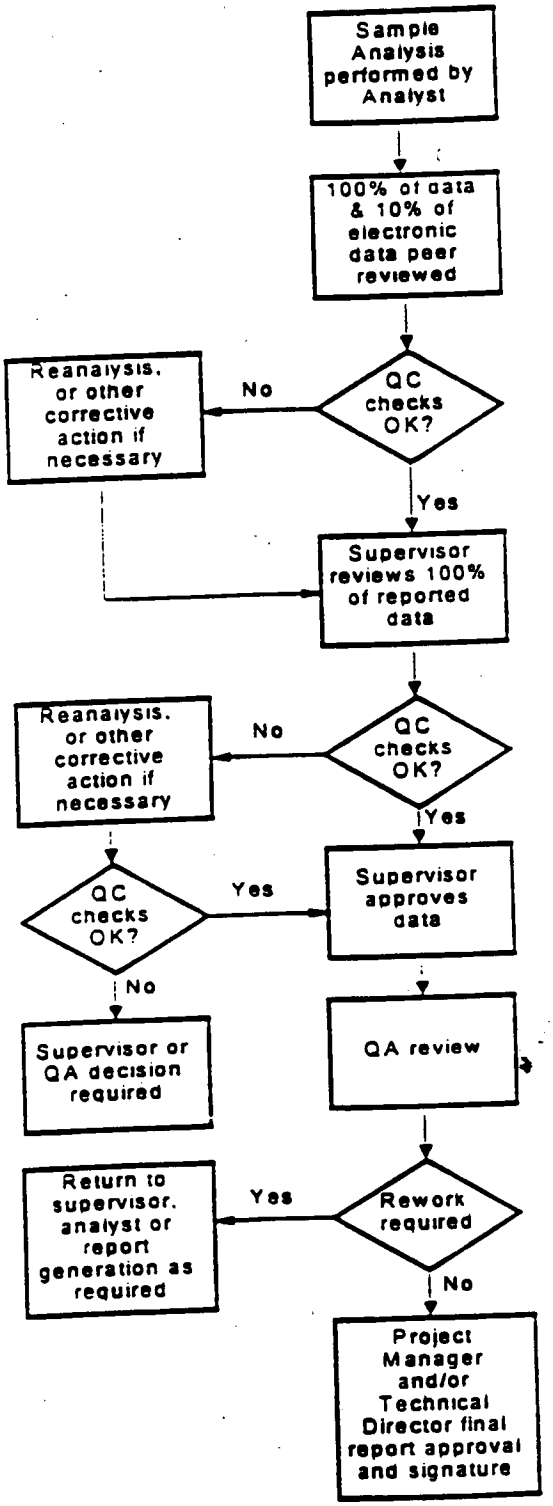
Laucks SOPs

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



-100 % maniual entries for transcription errors
-100% of manual calculations for accuracy
-10% spot check of computer calculations
-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 for compliance with Method or Project QC requirements
-Check for descriptions of deviations from Method or Project QC requirements
-Check reported values for dilutions
-Check reasonableness of data

-Check for reasonableness of reported data
-Check for completeness of the reported information
-Check for compliance with required QC practices
-Check for descriptions of deviations from Method or Project QC requirements
-Check the information for the report narrative

-Check for compliance with required QC practices
-Check for reasonableness of reported data
-Check for descriptions of deviations from Method or Project QC requirements
-Check for compliance with approved SOPs (periodically)

P:\SOP\QA_SOPS\RVW_CHT1.GFC 6/12/96

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

Written by: Harry Romberg, QA Officer Date: 5-13-98

Approved by: Kathy E Kreps, Laboratory Director Date: 5-18-98

Controlled Document

No. 20 Assigned to: Tetra

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EXAMPLE LOGBOOK REVIEW LABEL 11

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 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. This document applies to all personnel involved in the preparation, control and use of laboratory notebooks.

1.1.2 More specific instructions for maintaining logbooks can be found in pertinent SOPs, such as LTL-1007 "Maintaining Instrument Records and Logbooks" or LTL-1005 "Analytical Balances" or others specific to other laboratory operations.

1.2 Purpose

1.2.1 The purpose of this SOP is to define the practices used to maintain control and use of laboratory logbooks. This SOP is not intended as a specific description of any particular logbook type but covers the practices that must be in place for all logbooks employed at Laucks.

1.3 Definition of Terms

1.3.1 Logbook - Any bound or unbound document that forms a record of activities and pertinent data regarding an activity including but not limited to maintenance logs, standards logs, reagent chemical logs, analysis logs including instrument outputs (computer generated or strip chart recordings), balance and temperature logs, or any other regularly maintained record of activity.

2. Requirements

2.1 Control

2.1.1 Documents that specify quality requirements or activities affecting quality or evidentiary activities shall be controlled to ensure that correct documents are being used and properly archived when completed.

2.2 Maintenance

2.2.1 Quality assurance records (logbooks) shall be compiled and maintained in accordance with approved procedures.

2.3 Monitoring

2.3.1 Logbooks should be periodically monitored to ensure they are being properly maintained and information is being correctly recorded. Standard logbooks and run logs should be monitored at least semiannually by group supervisors or their designees. Maintenance and other logbooks need only be reviewed annually, unless previous review has demonstrated inadequacies in the logbook which require more frequent monitoring.

3. Responsibilities

3.1 Quality Assurance Manager

- Maintain the logs for control of laboratory notebooks and provide control numbers and labels as required.
- Approve format and proposed content of laboratory notebooks; minor changes to pre-printed forms do not need QA approval as long as their basic content does not change.
- Maintain master copies of notebook pages (in instances where pre-printed pages with a specific format are used); this may be in electronic or hardcopy form or both.
- Monitor satisfactory implementation of the requirements of this SOP

3.2 Responsible Supervisor

- Determine the format and content of notebooks used in their respective areas.
- Ensure that QA has been provided with an electronic version of all pre-printed logbook pages in order that they are later available for reprinting or editing.
- Ensure that all laboratory notebooks are properly labeled, including the appropriate control number.
- Ensure that personnel are adequately trained in the proper use of laboratory notebooks
- Periodically review laboratory notebooks to verify satisfactory implementation of the requirements of this SOP. Standard logbooks and run logs should be monitored at least semiannually by group supervisors or their designees. Maintenance and other logbooks need only be reviewed annually, unless previous review has demonstrated inadequacies in the logbook which require more frequent monitoring. This activity may be assigned to another individual but should not be the same individual who regularly completes the log itself.

3.3 Analyst

- Ensure that they are using the appropriate logbook and understand how to properly fill in the required fields.
- Ensure that any new logbook has been given a logbook number by QA before beginning to use it.
- Ensure that the logbook is clearly identified with an instrument ID and purpose or other appropriate title which will enable the analyst to easily identify the logbook.
- Ensure that if pre-printed logbook pages need to be modified, the modifications are approved by their supervisor and that an electronic copy or, if requested an original hardcopy have been provided to QA.

4. Procedure

4.1 Notebook Structure

4.1.1 Laboratory notebooks may be either bound or unbound as described below. Most logbooks should be bound in some fashion but it is recognized that this is not always possible, such as for vendor service records. These records may be stored in 3-ring binders or other suitable notebooks.

4.1.2 In some instances, logbooks may be created from instrument printouts or other pages that do not lend themselves to being pre-bound. In these instances, the log sheets may be stored in a 3-ring binder or other storage until enough sheets have been accumulated to have them bound with the laboratory comb binder.

4.1.3 All logbooks whether bound or unbound must be controlled by QA as designated by the appropriate QA Book Number label (see example in Appendix 1).

4.1.4 Bound notebooks shall conform to the following:

- Where feasible, binding will be of a type that will make the removal and reinsertion of pages readily noticeable.
- If pre-printed and bound, all pages will be sequentially pre-numbered. If the format of the notebook permits the use of the reverse side of the pages, both sides of each page will contain a sequential page number.

- Each page of the pre-printed bound notebook will contain, as a minimum, the laboratory name, logbook title, and sequential page number. Other elements may also be necessary for any specific logbook.

4.1.5 Unbound notebooks shall conform to the following:

- Unbound pages will be contained in a binder or folder that provides protection from damage.
- Each unbound page will contain a unique identifier (e.g., run number/date). For identification purposes, a continuous printout on fanfold computer paper requires only one identifier unless the sheets are separated.
- As noted above in 4.1.2, some unbound logbooks may eventually be bound if practical.

4.1.6 All notebooks will contain the following information on the cover:

- Laboratory name, Laucks Testing Laboratories, Inc.
- Control number assigned by the Quality Assurance Officer
- The department to which the logbook was issued
- The use of the logbook (i.e. balance calibration, instrument run-log, etc.)
- The department book number or title uniquely identifying that book, as required to identify the specific use of the book. This may include an instrument number or other logbook ID (such as a standards logbook ID). This is in addition to the QA logbook number.
- Start Date, the date on which the first entry was made
- End date, the date on which the last entry was made

4.2 Control of Logbooks

4.2.1 The QA Officer will maintain a master log of laboratory notebooks that contains as a minimum, the following information:

- Unique control number for each logbook
- Logbook title, which should reflect the type of information to be entered.

- Department to whom issued, for accountability only. A logbook will generally be assigned to a work station or function, and in no way is a laboratory notebook to be considered a "personal" notebook.

- Date issued, for accountability only.

- Date closed, for accountability.

4.2.2 Master sheets for each logbook will be maintained by the QA Officer and will be utilized for producing notebooks when required.

4.3 Use of Laboratory Logbooks

4.3.1 The notebook is the basic document for recording information. Entries should be made into the notebook in real time, not written on scratch paper and transferred later.

4.3.2 Handwritten entries should be legible and entered in black or blue indelible ink.

4.3.3 Computer-generated data should be printed out and collected at appropriate times to represent the activities being recorded.

- Computer printouts may be either placed in unbound notebooks as described above, or inserted into bound notebooks.

- Computer printouts or other material inserted into bound notebooks must be securely fastened (tape is preferred) in such a way that removal and insertion of material can be determined readily.

4.3.4 When information from related activities is recorded in more than one notebook, provide adequate cross-reference information in all affected notebooks so that all pertinent data can be readily accessed.

4.3.5 Do not skip pages when entering data. For example, if data is not readily available for entry, do not leave space for later entry. Enter the data when it becomes available and provide adequate cross-references if required.

4.3.6 In cases where partial or complete pages must be left blank and not used, indicate the unused portion by placing a horizontal line at the beginning and end of the unused portion and connecting opposite ends of the horizontal lines with a diagonal, resulting in a Z-shaped figure. The individual striking out the blank area will initial and date the diagonal.

4.3.7 Errors or other changes must be deleted in a similar fashion or with a single-line cross-out which has been initialed and dated. **No erasures, overwriting, white-out or multiple-line cross-outs (blacking out) are acceptable.**

4.3.8 When pre-printed formats are used and all possible entries are not required, the remaining blanks may be struck out with a Z as described above, or entries such as N/A may be placed in the unused blanks.

4.3.9 The individual entering information into the notebook shall initial and date each page used, or in the case of logbooks with ongoing records which do not occupy the entire page, such as maintenance logs or balance logs, each individual entry.

4.4 Supervisory Monitoring of Laboratory Logbooks

4.4.1 Standard logbooks and run logs should be monitored at least semiannually by group supervisors or their designees. Maintenance and other logbooks need only be reviewed annually, unless previous review has demonstrated inadequacies in the logbook which require more frequent monitoring. This activity may be assigned to another individual but should not be the same individual who regularly completes the log itself.

4.4.2 Logbooks should be reviewed using the review items provided in Appendix II, although it is not necessary to actually document the review using this checklist.

4.4.3 Errors should be formally brought to the attention of the responsible individual through the use of Corrective Action Forms. If errors are correctable or items can be corrected for legibility problems, they should be corrected using the proper error correction technique.

4.4.4 Logbooks that have been reviewed are marked with a fluorescent yellow or other colorful label that looks similar to the label in Appendix III.

4.5 QA Monitoring of Laboratory Logbooks

4.5.1 The QA Officer will verify during periodic audit and surveillance activities that notebooks are properly completed and maintained. This will generally be done approximately annually as part of routine audits. This observation does not preclude the requirement for supervisory review.

Appendix I

Example QA Logbook Label

Laucks
Testing Laboratories, Inc.

QA Book No.: _____

Issued To: _____

Used For: _____

Dept. Book No.: _____

Start Date: _____

End Date: _____

Appendix II

Example Logbook Review Items to be Observed

- Have all pertinent fields been filled or marked not applicable (N/A)?
- Has empty space been crossed out properly initialed and dated?
- Have errors been corrected with single-line crossouts, initialed and dated (no obliterations or overwrites)?
- Are all entries clear and easy to read and comprehend?
- If calculations are involved, check several random calculations for error.
- If traceability is involved (as for standards) check several random entries to confirm that the logbook entries can be tracked back to the original entry.
- If standards log, observe some actual standards and compare them against logbook entries for accuracy.
- Are all handwritten entries initialed and dated?
- If the book is beginning to deteriorate, it should be repaired or retired and replaced.

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

Revised by: _____ Date: 8/25/97
Monica Carr, Organics Division Manger

Reviewed by: _____ Date: 8-25-97
Harry Romberg, QA Officer

Approved by: _____ Date: 8/25/97
Karen Kotz, Laboratory Director

UNCONTROLLED

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1. Introduction and Scope

1.1 Method Description

1.1.1 This SOP describes the integration process for chromatographic data, the procedures for manual integration, and the procedures for documenting manual integration.

1.1.2 Integration identifies peaks found in the data collected during data acquisition and characterizes them. The software uses the integrated peaks to determine the identity and quantity of compounds in the samples. The peak area, peak height, peak type, baseline, and retention time of each peak in a chromatogram are determined by integration. Some peaks, due to limitations of the software, will need to be manually integrated. The manual integration process must be documented. This documentation will include a brief description of why it was necessary, who did it, when was it done, and a hardcopy of the re-integrated peak.

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 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/LAS and EZChrom
2.1.2 GC/MS-Teknivent/EnviroQuant
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: 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). The manual integration must also be documented in the associated sample narrative. The supervisor 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: 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.

5.1.5 LAS - After changing the integration, print the chromatogram.

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 EZChrom Chromatography Data System, Scientific Software, Rev. 6.6, 1995.

6.1.4 Wisconsin DNR Newsletter, October 1994.

6.1.5 SOP# LTL-1018, Standard Operating Procedure for Review and Approval Practices for Validatable Packages.

6.1.6 SOP # LTL-8005, Standard Operating Procedure for GC Gas/BTEX Data Review.

6.1.7 SOP #LTL-8004, Standard Operating Procedure for GC Volatiles Data Review.

6.1.8 SOP #LTL-8001, Standard Operating Procedure for GC Hydrocarbons Data Review.

6.1.9 SOP #LTL-8301, Standard Operating Procedure for HPLC Aromatics 8310 Data Review.

6.1.10 SOP #LTL-8302, Standard Operating Procedure for HPLC Ordnance 8330 Data Review.

6.1.11 SOP #LTL-8201, Standard Operating Procedure for GC/MS VOA Data Review.

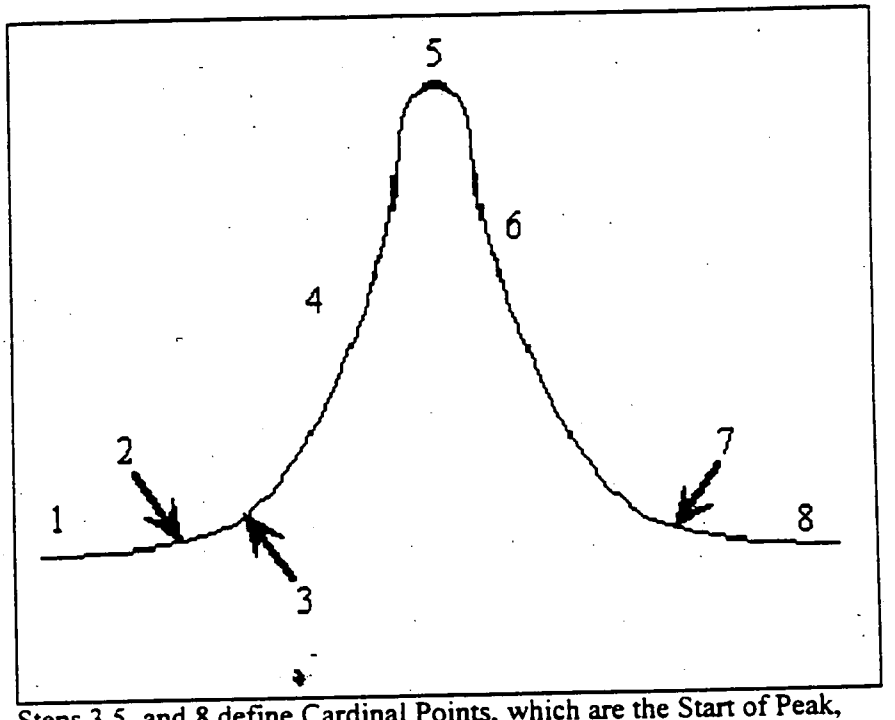
6.1.12 SOP #LTL-8202, Standard Operating Procedure for GC/MS Semivolatile Data Review.

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.

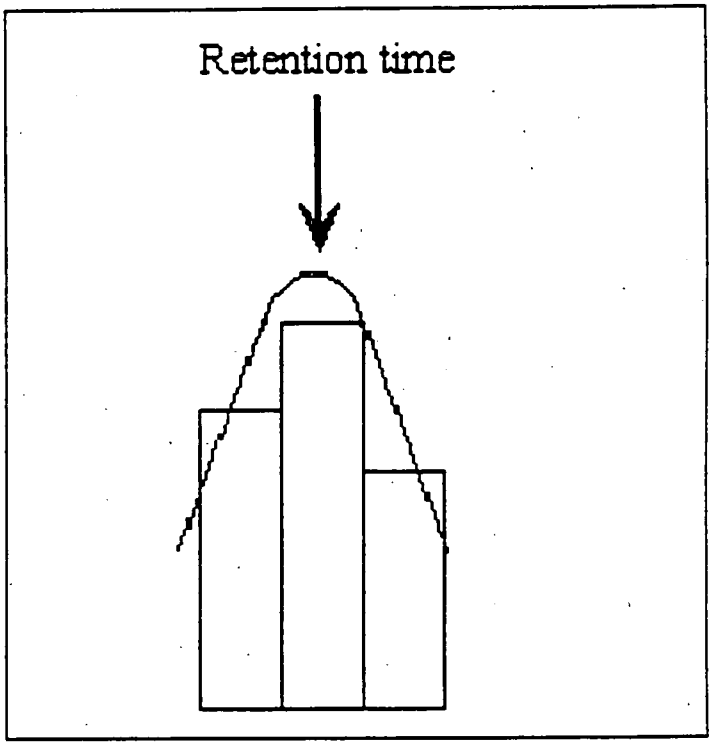
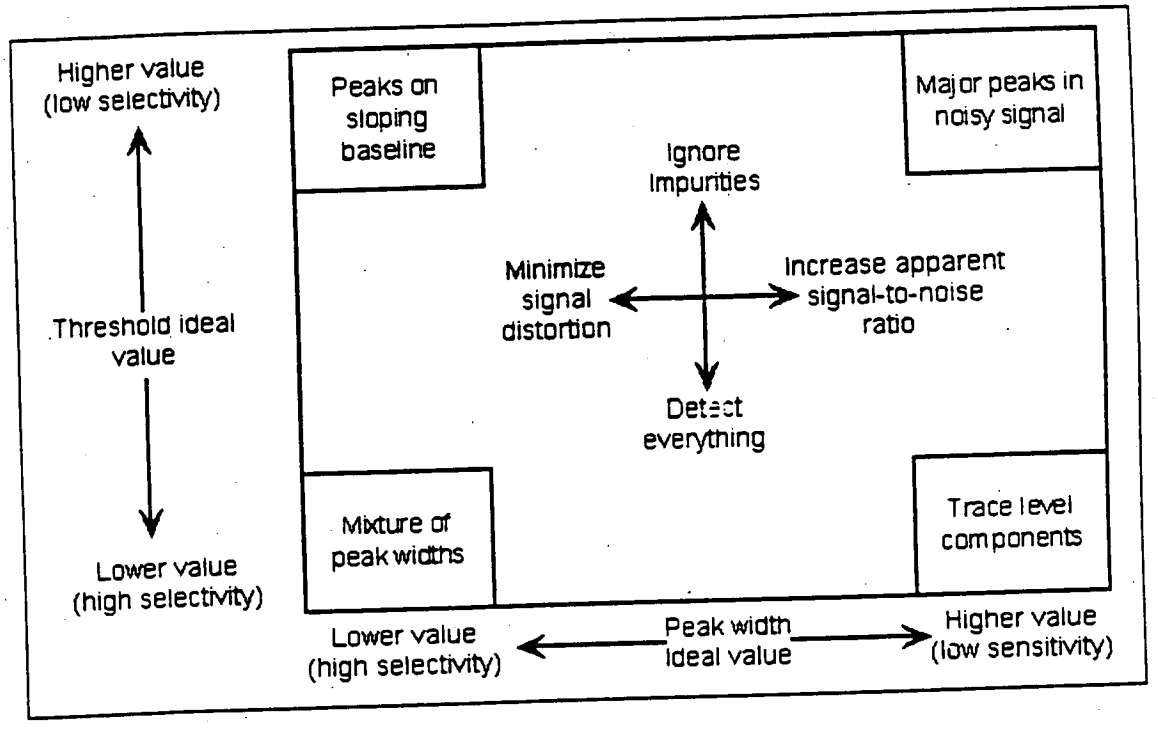
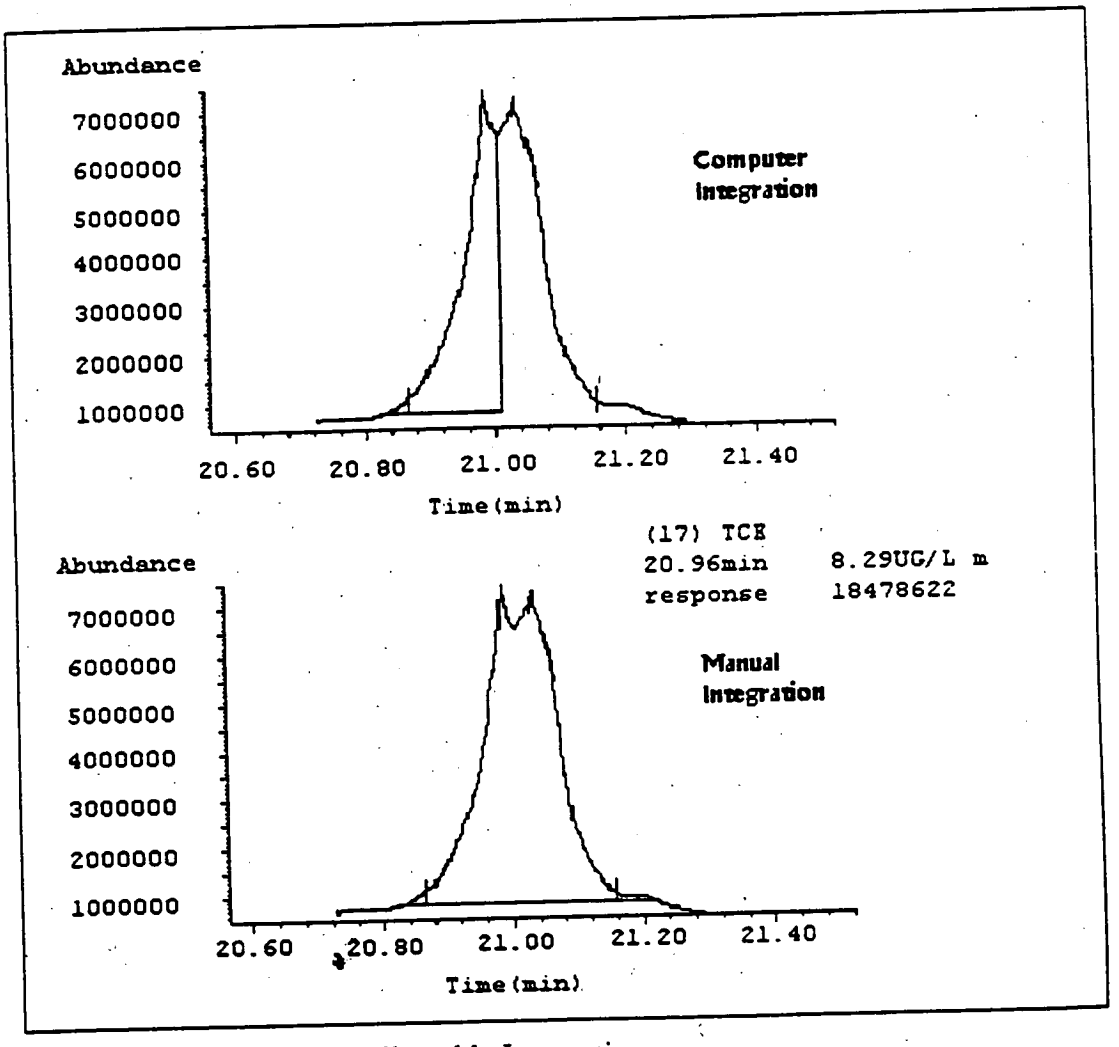


Fig 3.


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

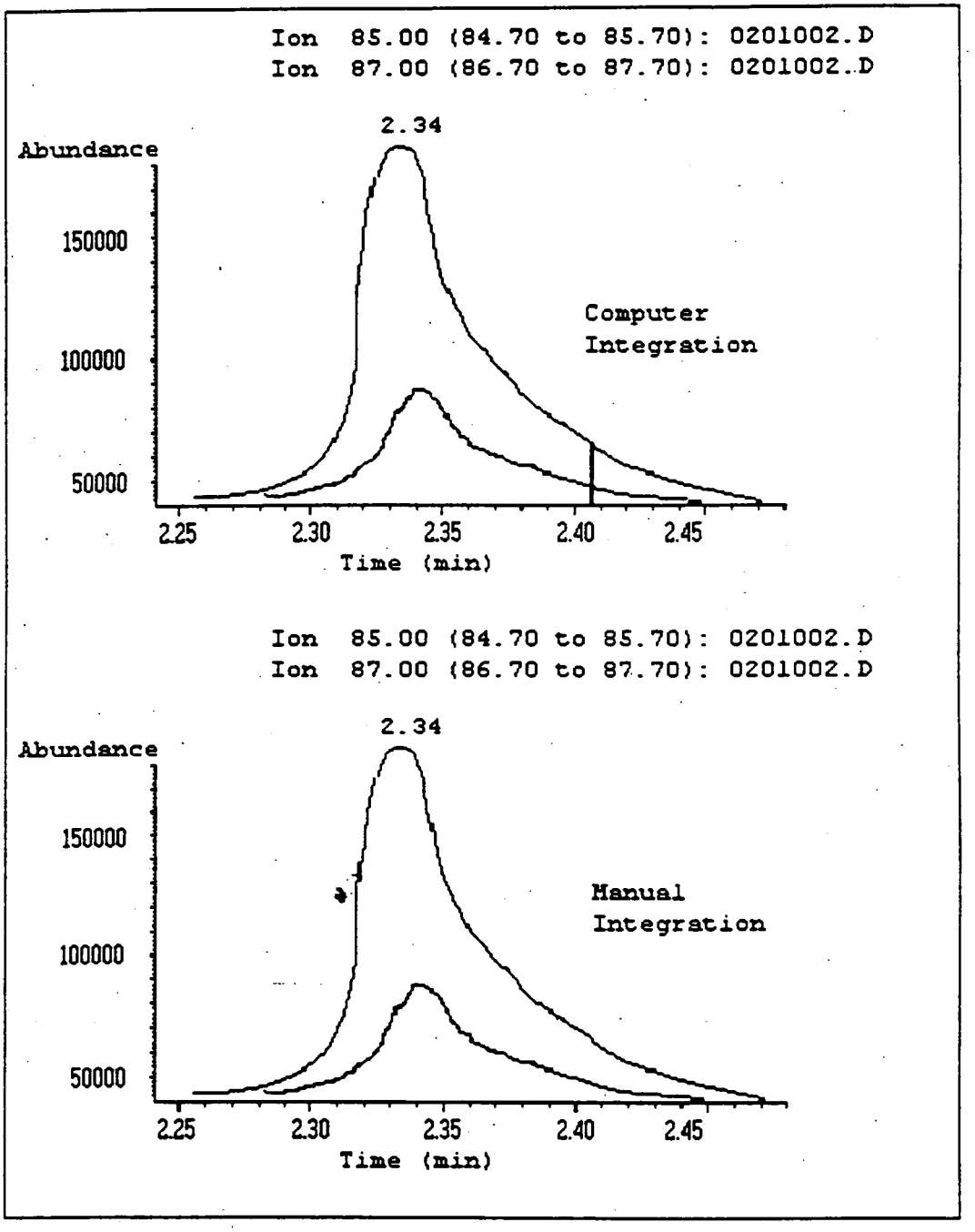
Attachment 1



Allowable Integration

Split Peaks

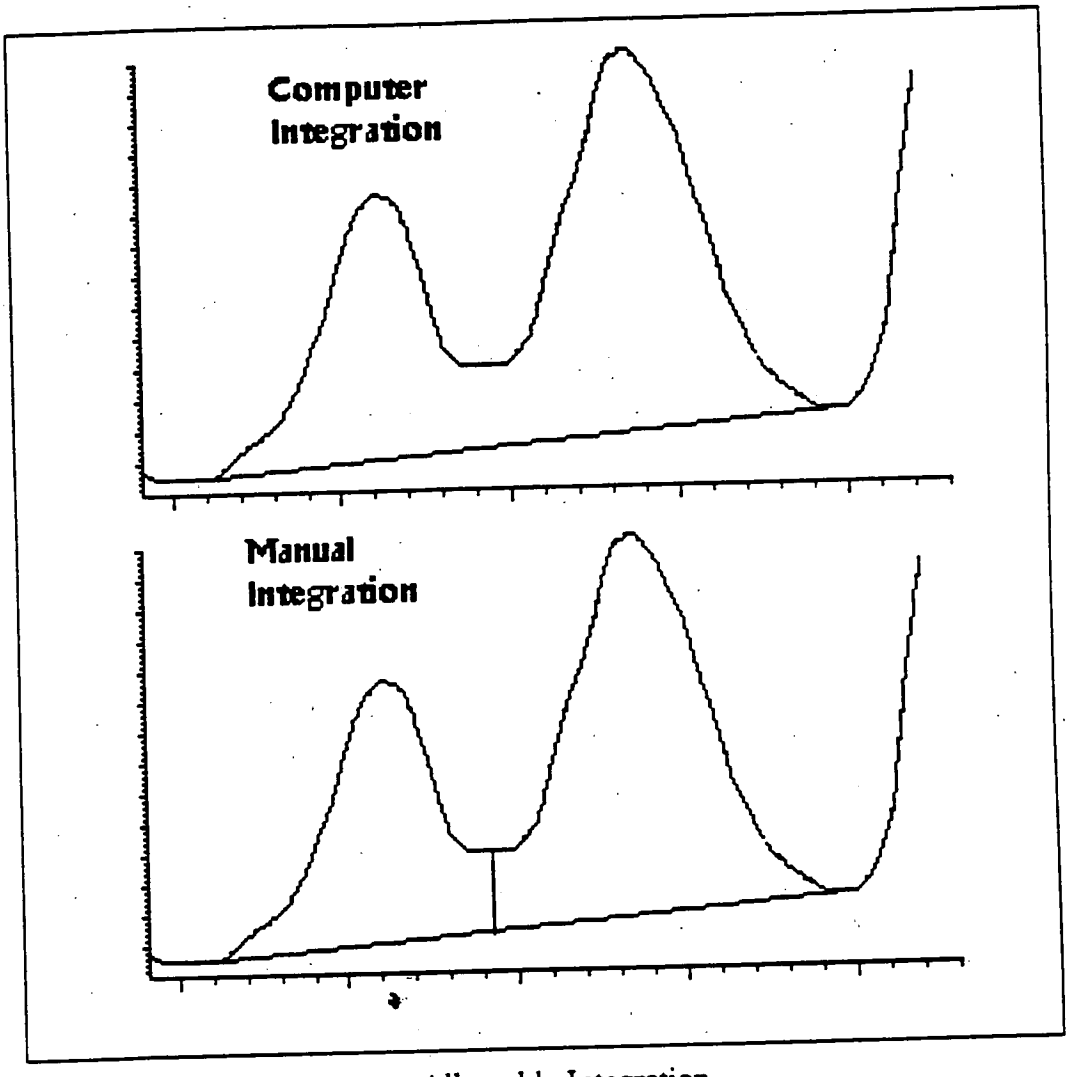
Attachment 2



Allowable Integration

Tailing

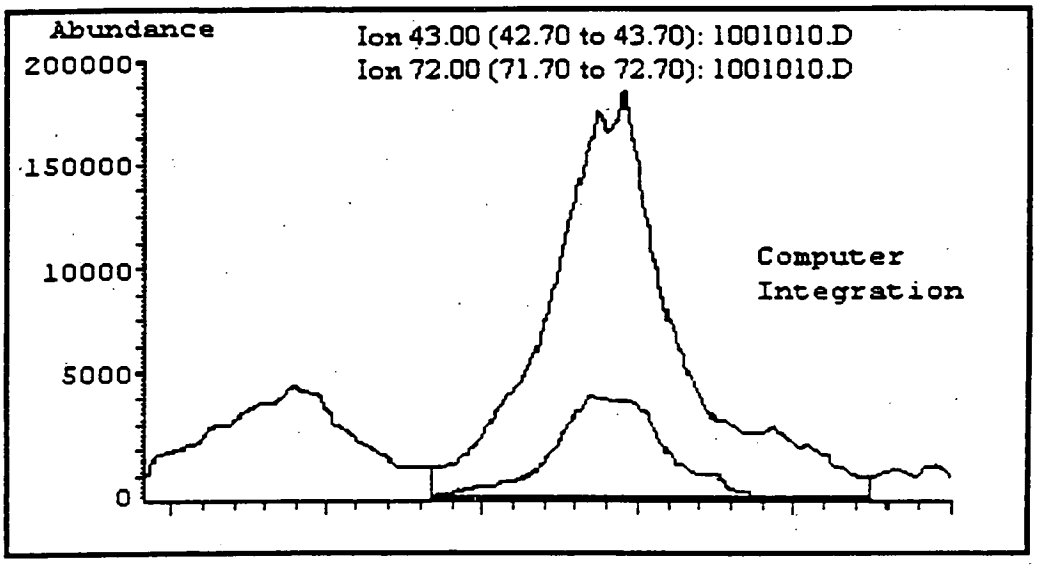
Attachment 3

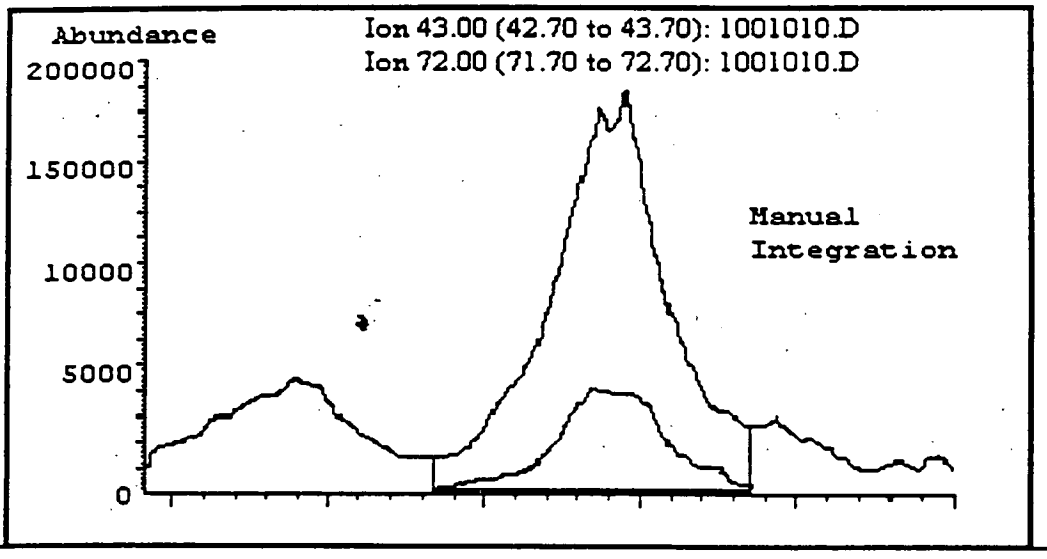


Allowable Integration

Retention Time Shift - Mis-Identification - Merged

Attachment 4

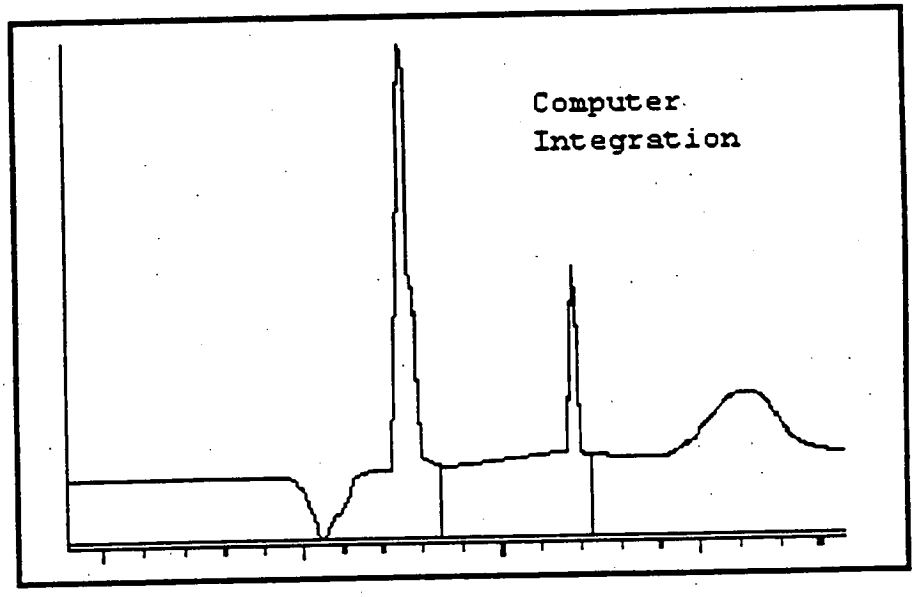


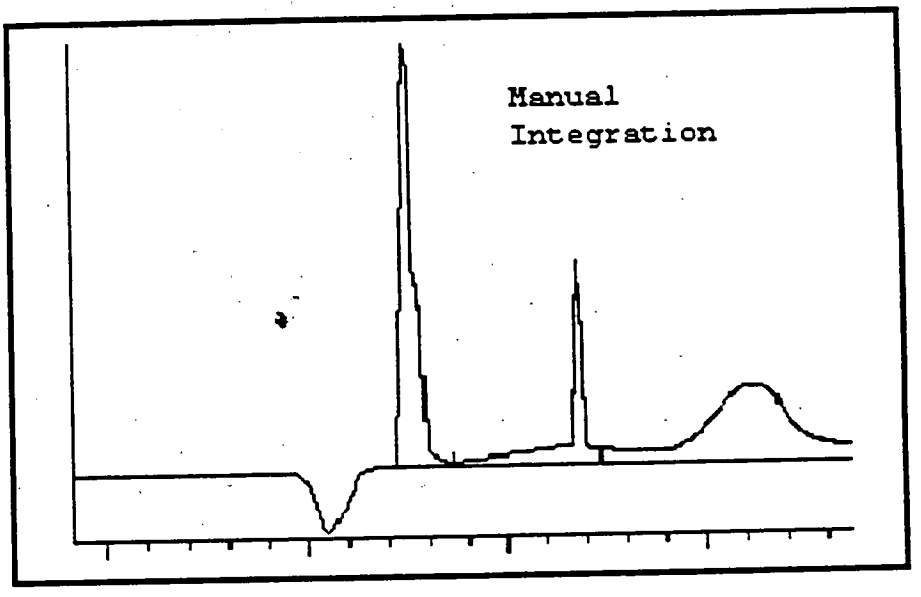


Allowable Integration

Secondary or Qualifier Ions

Attachment 5

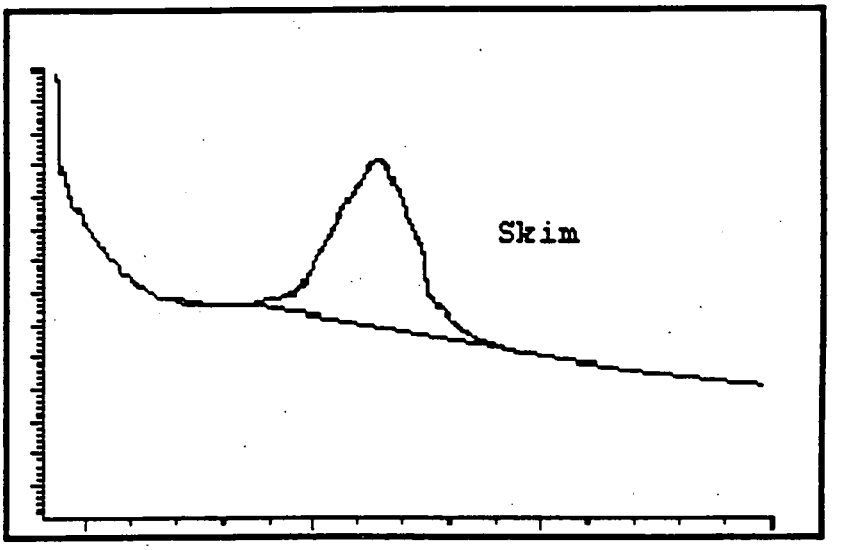


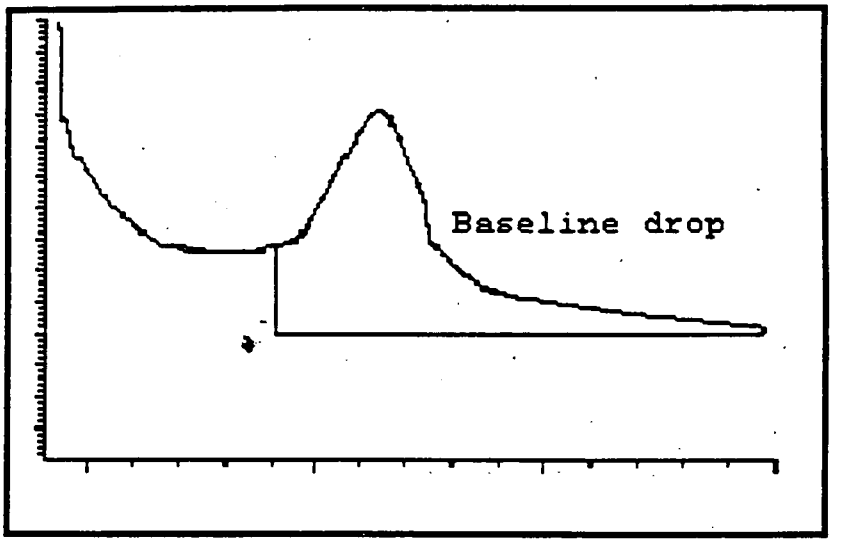


Allowable Integration

Baseline Shift

Attachment 6

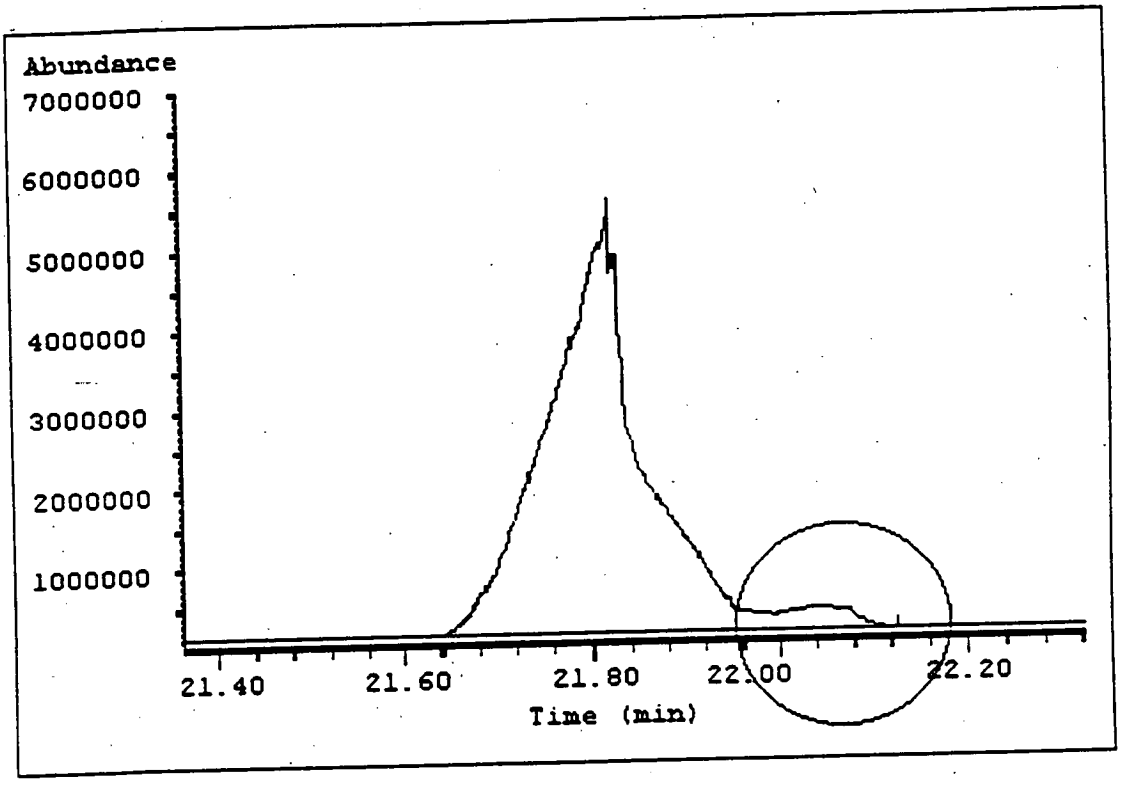




Allowable Integration

Skimming vs Baseline Drop

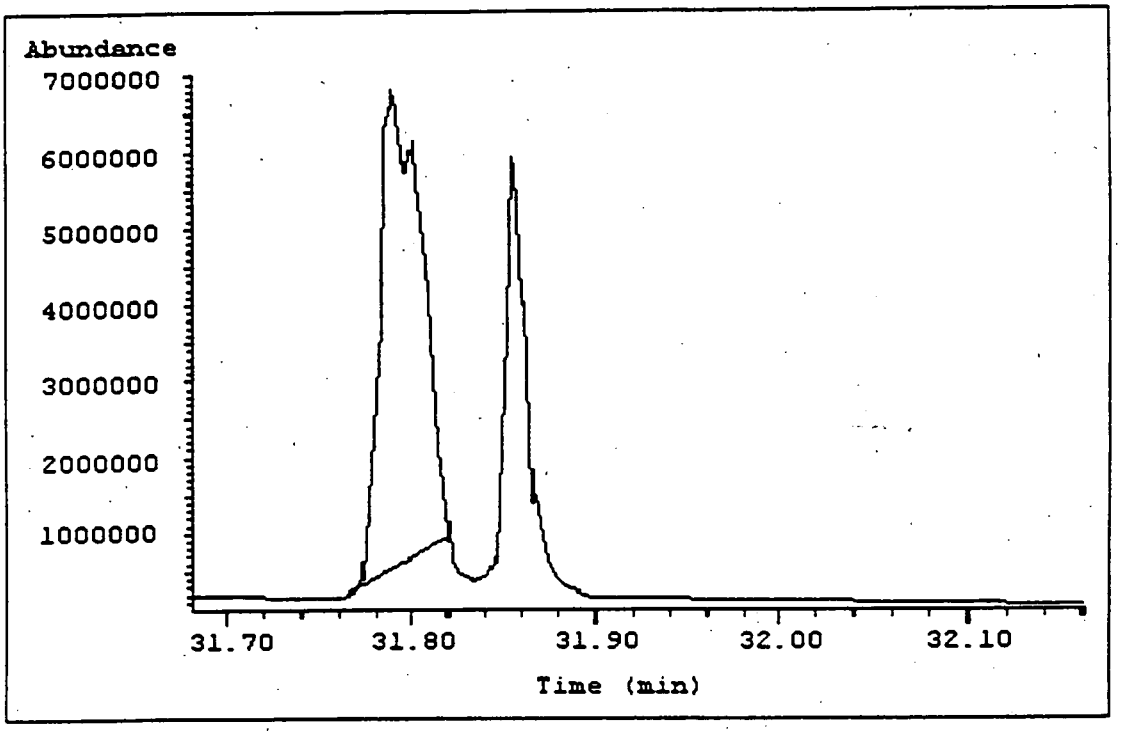
Attachment 7



Improper Integration

Adding Area by Including Other Peaks

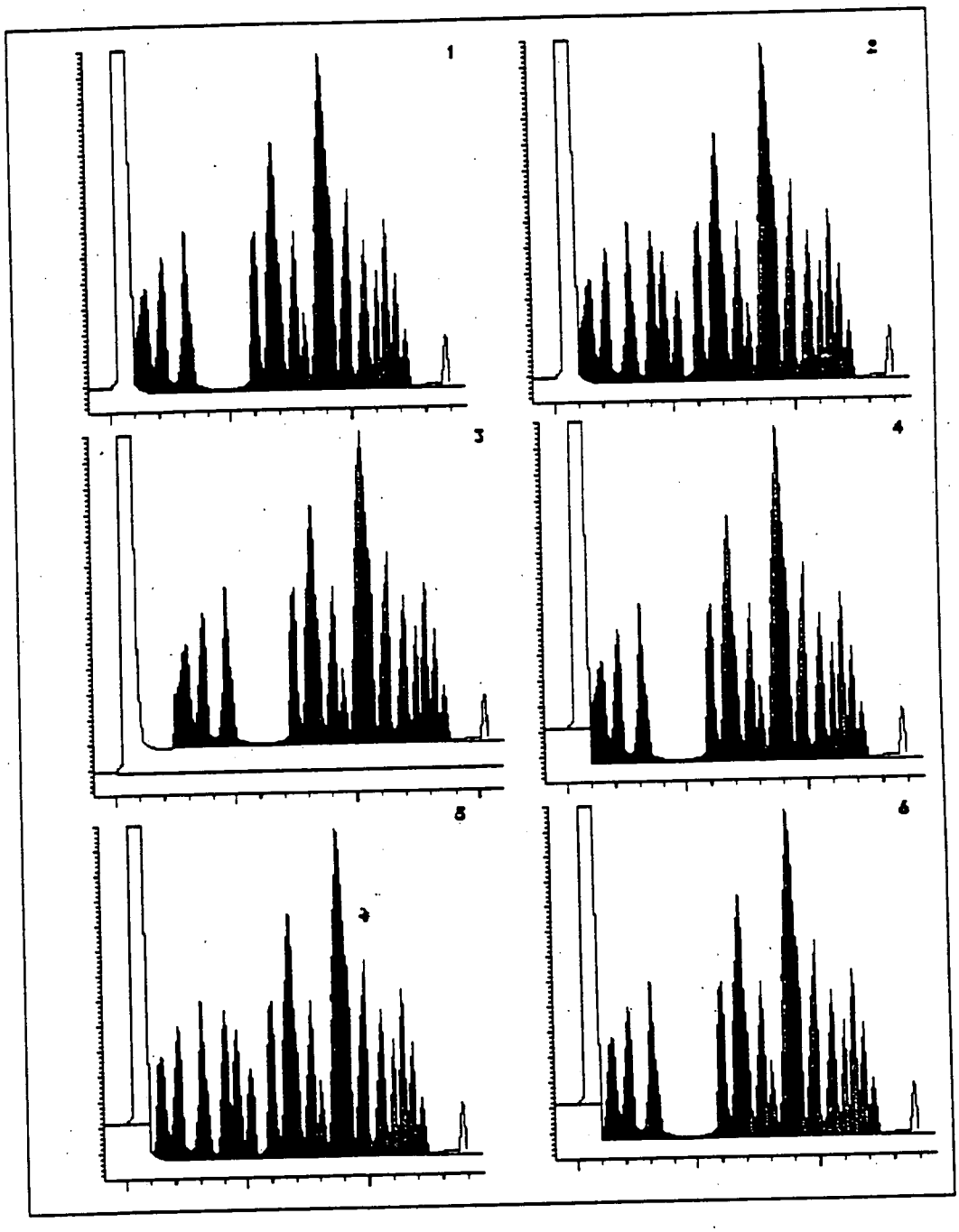
Attachment 8



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

Written by: Harry Romberg Date: 6-2-98
Harry Romberg, Quality Assurance Officer

Approved by: Kathy Kreps Date: 6-2-98
Kathy Kreps, Laboratory Director

Controlled Document
No. 20 **Assigned to:** Tetra

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1. Introduction and Scope

1.1 Method Description

1.1.1 The purpose of this SOP is to describe the laboratory waste disposal scheme currently in place at Laucks. The primary waste streams described include solvents, PCB oil wastes, COD and TOC waste and soil samples. 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 the 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.

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 TOC 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 and TOC wastes, for instance, contain high levels of mercury salts. Direct contact should be avoided through the use of proper clothing and eye wear, even if no immediate danger is obvious. In the case of volatile solvents and other materials, handling should be done in a well-ventilated area and the exposure to vapors minimized. Where strong fumes are unavoidable, a carbon-filter or other respirator should be worn.

3.1.4 All people who handle waste products or the original reagents should be aware that the laboratory provides safety equipment and has a file containing Material Safety Data Sheets (MSDSs) on all laboratory chemicals in support of OSHA and other safety programs.

4. Operation procedures

4.1 Operations Appropriate to All Collection Areas

4.1.1 Land Disposal Restriction Forms (LDRs), manifests and other paperwork are not extensively discussed in this SOP because the disposal vendor deals with this aspect of the paperwork. It will only be necessary for the person who will be asked by the vendor to sign these forms (usually QA) to check that the information on the forms is accurate and to sign the form.

4.1.2 All waste requiring a Hazardous Waste disposal sticker and manifest will be marked with one of two EPA Hazardous Waste Site numbers. All waste transported from the 921 facility will be numbered WAD981762024 and all waste transported from the 940 facility will be numbered WAD027446608.

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.

4.1.4 All collection drums **must** be marked with an appropriately filled out Hazardous Waste sticker (see Appendix A). It is only necessary for Laucks staff to fill in the date that collection was **started** and the contents of the drum in the appropriate space. Hazardous Waste cannot be accumulated for longer than 90 days before it must be disposed. Therefore, do **not** mark the date on the drum until collection is started so as to maximize the allowable time until disposal. This 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 In addition, corrosive and flammable waste collection drums must have a sticker which indicates their corrosive or flammable nature (see Appendix B).

4.1.6 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 point that collection was **started**. 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.

4.1.6.1 The one variation from the above rule is the TOC waste drum. This drum is not a satellite collection point but is actually the catch drum for the waste directly from the instrument. It will be disposed as soon as possible after it has reached capacity.

4.1.7 When collection drums are full or the 90-day limit is approaching, the Quality Assurance (QA) Department must be notified. The preferred lead-time for pickup is 10 working days so QA should actually be contacted 80 days after collection is

initiated. This department, at the time of this writing, is responsible for contacting the appropriate approved transporter and insuring proper disposal takes place.

4.1.8 All questions or concerns regarding hazardous waste operations should first be directed to QA who will determine the appropriate course of action.

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 is in the 921 facility (Extractions) 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 in Extractions when full. 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.

4.2.3 When new materials are collected in the primary drum, a Hazardous Waste sticker should be affixed with an initial collection date. The Hazardous Waste stickers should be marked with a DOT shipping name of **"Waste Flammable Liquids"**.

4.2.4 Although methylene chloride is non-flammable, other components of these waste drums may be highly flammable. Thus, all of the waste solvent containers must be labeled as flammable.

4.2.5 At least 2 inches of headspace must be left between the top of the liquid and the top of the drum to allow for expansion.

4.2.6 When 3 or more full 55 gallon drums of this waste have been accumulated or 80 days have passed since the beginning of collection of the oldest accumulated drum, QA must be contacted to arrange for transport and disposal.

4.2.7 At the time of this writing, Laucks uses Laidlaw Environmental 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 as corrosive with the appropriate sticker as previously noted.

4.3.4 The waste not held for more than 90 days from initial collection (after pouring from the reaction tubes) until transportation for disposal. After 80 days have passed since the beginning of collection, QA must be contacted to arrange for transport and disposal within the allowable timeframe.

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 Laidlaw Environmental as the facility of choice for handling this waste stream. This vendor can be changed at the discretion of QA on either a one-time or ongoing basis.

4.4 Total Organic Carbon (TOC) Waste

4.4.1 The primary constituents of this waste are mercury, potassium persulfate, nitric acid, and water. This waste is collected directly from the instrument into a waste container beneath the instrument.

4.4.2 As this is a continuous process, Laucks does not begin the 90 day clock before disposal is required until this container is full. However, the container must be marked with a corrosive sticker. The Hazardous Waste sticker, in this case, must be dated as soon as the container is full and affixed at that time. The Hazardous Waste Sticker should be marked with a DOT shipping name of "**Waste Corrosive Liquids, Acidic, Inorganic**".

4.4.3 At least 2 inches of headspace must be left between the top of the liquid and the top of the container to allow for expansion.

4.4.4 As soon as the container is full, the QA department must be notified to arrange for disposal. This waste stream will generally not be held in storage for very long after collection.

4.4.5 At the time of this writing, Laucks uses Laidlaw Environmental as the vendor of choice for handling this waste stream. This vendor can be changed at the discretion of QA on either a one-time or ongoing basis.

4.5 Soil Sample Disposal

4.5.1 State law allows a laboratory to store samples and other materials indefinitely, until they are considered waste and disposed. After that time, from the date of first accumulation, a 90 day timeframe is allowed before disposal must occur. Thus, soils

should not be disposed of until enough have been accumulated to fill at least one 55 gal. drum.

4.5.2 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 appropriate marked with the assigned drum number. This will enable the laboratory to track which samples were disposed in which drum.

4.5.2.1 The drums should be marked with a year, location from which they originate, and sequential number. Thus drums for which accumulation began in 1998 from the extractions laboratory would be marked 98-921-01. The -01 being a sequential number that would be incremented with each additional drum -02, -03, etc. throughout 1998. A drum from the main lab would be designated 98-940-01, etc.

4.5.2.2 When samples are transferred from the storage locations to the drums. the Secure Storage Custody Log must be marked to indicate into which drum they were disposed. This should include any bottle identifiers, if necessary to identify just what was disposed. Thus, it will be necessary for personnel disposing of samples to check the drums to make sure there is enough room for the designated samples. Soil samples will generally have their lids removed and disposed in the regular garbage. The 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.

4.5.2.3 When the drums are disposed, it will be necessary for the laboratory representative who signs the manifest to mark the drum identity on the manifest, although this only needs to be on the laboratory copy if the transporter does not want this information to appear on their copy of the record.

4.5.3 QA must be notified 80 days after accumulation has begun in order to arrange for disposal in a timely manner. If samples are not disposed until there is enough to fill a drum, this timeframe is not of major concern because there are always Hazardous Waste pickups scheduled within any 90 day time period.

4.5.4 The only stickers these drums must have is the Hazardous Waste sticker with the date accumulation was started clearly marked. The Hazardous Waste Sticker should be marked with a DOT shipping name of "**Waste Environmentally Hazardous Substances**".

4.5.5 At the time of this writing, Laucks uses Laidlaw Environmental as the vendor of choice for handling this waste stream, although this could be changed at the discretion of QA on either a one-time or ongoing basis. This vendor incinerates these soils prior

to landfilling which should dispose of any organic materials, including labels, oily material and other hazardous organic substances.

4.6 PCB Oil Waste Disposal

4.6.1 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.

4.6.2 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.

4.6.3 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.

4.6.4 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.

4.6.5 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 produce all of the required paperwork and insure all of the appropriate container markings (stickers, etc.) are in place prior to shipment. Since Laucks' waste streams are consistent from time to time, our vendors already have the information required to properly fill out the paperwork and Hazardous Waste stickers.

5.1.1.1 The paperwork includes the manifests, land disposal restriction forms and other shipping paperwork. Thus the only requirements of the laboratory are to insure the paperwork is accurate and to sign the appropriate forms.

5.1.2 After the waste has been transported to the disposal or accumulation facility, a signed manifest is returned to the laboratory. This is kept with the permanent record.

5.1.3 All certificates of disposal later provided by the disposal vendor are also associated with any waste shipment and kept with the permanent record.

5.1.4 All records are retained for at least 5 years from the date of shipment of the waste.

5.2 Annual Reporting Requirements

5.2.1 The laboratory must file an annual report with the Washington Department of Ecology (WDOE) for legal and tax purposes. This report is due on March 1 each year. Reports are filed for both the 940 and the 921 facilities (both EPA ID numbers). All waste transported from the 921 facility will be numbered WAD981762024 and all waste transported from the 940 facility will be numbered WAD027446608.

5.2.1.1 The only exception to this reporting requirement is the reporting of the PCB waste oil which is a federally regulated waste and is thus not reported to the WDOE.

5.2.2 The format of this report is defined by WDOE in books provided to the laboratory several months in advance of the due date. Details of this report are not provided in this SOP.

5.2.3 In addition, as part of a WDOE program to reduce hazardous waste in general, Laucks files an annual pollution prevention plan update in September of each year. This report is more loosely defined and the only major requirement is that it be filed. Details of this report are not part of this SOP.

Appendix I

Hazardous Waste Sticker

See directions in this SOP for proper filling out of this sticker.

HAZARDOUS WASTE

FEDERAL LAW PROHIBITS IMPROPER DISPOSAL

IF FOUND, CONTACT THE NEAREST POLICE, OR
PUBLIC SAFETY AUTHORITY, OR THE
U.S. ENVIRONMENTAL PROTECTION AGENCY

PROPER D.O.T.
SHIPPING NAME_____ UN OR NA#_____

GENERATOR INFORMATION:

NAME_____

ADDRESS_____

CITY_____ STATE_____ ZIP_____

EPA
ID NO._____

EPA
WASTE NO._____

ACCUMULATION
START DATE_____

MANIFEST
DOCUMENT NO._____

HANDLE WITH CARE!

CONTAINS HAZARDOUS OR TOXIC WASTES

STYLE WM-6

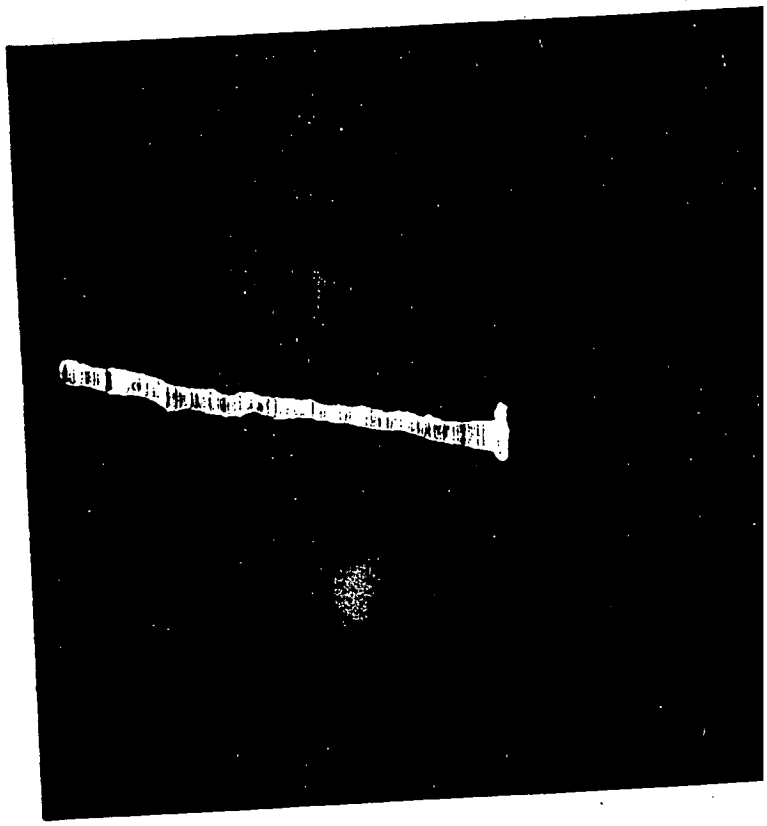
Printed by LABELMASTER, Div. of AMERICAN LABELMARK CO., INC., CHICAGO, IL 60646

Appendix II

Corrosive and Flammable Stickers

8-HML

Published by J. J. KELLER & ASSOCIATES, INC.
Neenah, WI 54956-0368 ☎(414)722-2848



Extraction Method for Ordnance Compounds in Water (8330)

Method # LTL-3077

October 29, 1998

UNCONTROLLED

Revision Number: #3

Written by: B. O'Neill Date: 10-29-98

Reviewed by: Harry Romberg Date: 10-29-98

Approved by: Kathy Kreps Date: 10/29/98

1. **PURPOSE** - In this method the ordnance compounds are extracted from neutral water with acetonitrile. The extract is salted-out and analyzed by HPLC.

2. **SAFETY** - During the conduct of this method, the extractionist will be exposed to a variety of reagent chemicals and solvents. The health effects of these various chemicals may be ascertained by reading the material safety data sheets (MSDS) available in the general files. Additionally, the samples by their very nature, may contain significant levels of hazardous materials. It is incumbent on each extractionist to exercise due care and caution while executing this method. The company will provide any protective equipment or clothing needed to assure employee safety.

3. **REAGENTS**

3.1. All reagents shall be of AR grade or better.

3.2. All solvents shall be distilled in glass unless otherwise indicated.

3.3. The following special reagents shall be prepared:

3.3.1. Sodium Chloride - prepared by muffling AR grade sodium chloride for four hours at 400°C.

3.3.2. Salt Water - prepared by weighing 325 grams muffled sodium chloride, dissolving in DIW and bringing to 1000 ml.

3.3.3. Surrogate solution prepared in methanol:

1,2-Dinitrobenzene	80 ug/ml

3.3.4. Matrix spiking (MS) solution prepared in methanol:

1,3-Dinitrobenzene	40 ug/ml
2,4-Dinitrotoluene	40 ug/ml
2,6-Dinitrotoluene	40 ug/ml
HMX	40 ug/ml
RDX	40 ug/ml
Nitrobenzene	40 ug/ml
2-Nitrotoluene	40 ug/ml
3-Nitrotoluene	40 ug/ml
4-Nitrotoluene	40 ug/ml
Tetryl	40 ug/ml
TNT	40 ug/ml
1,3,5-Trinitrobenzene	40 ug/ml
2-amino-4,6-dinitrotoluene	40 ug/ml
4-amino-2,6-dinitrotoluene	40 ug/ml

3.3.5. Special Matrix spiking solution for PETN/NG in methanol:

PETN	25 ug/ml
Nitroglycerin	50 ug/ml

3.3.6. Special Matrix spiking solution for 6 additional compounds in methanol:

2,4-DA-6-NT	40 ug/ml
2,6-DA-4-NT	40 ug/ml
3,5-DNA	40 ug/ml
MNX	40 ug/ml
TNX	40 ug/ml
4,4'-TN-AZOXY	40 ug/ml

4. EQUIPMENT

4.1. Magnetic stirrer with stirring bar

4.2. Standard laboratory glassware to include:

4.2.1. 1000 ml volumetric flask and various sizes of graduated cylinders

4.3. All glassware to be rinsed as follows, prior to use:

4.3.1. Technical grade acetone (if the glassware is wet).

4.3.2. Triple rinsed with methylene chloride.

4.4. Volumetric measurements are to be made with a calibrated fixed or adjustable volume microdispenser and individually calibrated vials.

4.5. PTFE 0.45µm syringe filter.

5. **QUALITY CONTROL** - The normal level of quality control will consist of blanks, blank spikes, matrix spikes and matrix spike duplicates (MS and MSD). This is performed on a per batch basis to include no more than 20 samples. The level of quality control will be indicated to the extractionist at the time the job is assigned. These samples serve to provide a measure of the recovery efficiency for the analyte and to provide data for statistical evaluation of the sample. In those instances that a client requires additional or different quality control measures, the extractionist will be directed accordingly in writing.

6. METHODOLOGY

6.1. Sample Extraction

6.1.1. Mix sample thoroughly in its original container.

6.1.2. Measure 770 mls sample into a 1000 ml volumetric flask placed on a magnetic stirrer.

6.1.3. Prepare two additional aliquots of one sample if Quality Control is required.

6.1.4. Prepare a blank and blank spike with DIW.

6.1.5. Pipet 250 ul surrogate solution to each flask.

6.1.6. Pipet 500 ul matrix spiking solution to each of the QC flasks. (When indicated, use 500 ul special matrix spiking solution.)

6.1.7. Add 251.3 gm sodium chloride to the flask with the stirring bar at maximum speed and stir until sodium chloride is completely dissolved.

6.1.8. Add 164 mls acetonitrile to each flask and stir for 15 minutes.

6.1.9. Turn off the stirrer and let set for 10 minutes.

6.1.10. Remove acetonitrile with a Pasteur pipet and transfer to a 100 ml flask.

6.1.11. Add 10 ml acetonitrile to the sample and stir for 15 minutes.

6.1.12. Turn off stirrer and let set for 10 minutes.

6.1.13. Transfer acetonitrile to flask with extract from step 6.1.10. Discard sample in 1000 ml flask.

6.1.14. Add 84 mls salt water to extract in flask.

6.1.15. Stir for 15 minutes and let stand for 10 minutes.

6.1.16. Transfer acetonitrile to a 10 ml graduated cylinder.

6.1.17. Add 1.0 ml acetonitrile to flask and repeat steps 6.1.15 and 6.1.16.

6.1.18. Record total final volume to 0.1 ml.

6.1.19. Transfer final extract to a 4.0 ml **amber** vial. Any remaining extract may be discarded. If extract is cloudy or turbid, filter through a PTFE 0.45µm syringe filter into amber vial.

6.1.20. Label the extract and deliver to 940.

6.1.21. Complete all paperwork and bench sheet. Bench sheet to include date and time of transfer to 940 and extract location. Clip the T-card on the folder and place in GC room extraction folder box. The file folder color will be red and the blank name will be ____HOR.WL_.

7. **ANALYSIS TIME** - Based on experience in the laboratory, it is anticipated that a single sample may be completed in approximately 3 hours. If it is possible to batch similar samples, it is expected that about ten samples could be completed in approximately 8 hours. About one hour of actual hands on time will be expended for a single sample by the extractionist. These approximate times are based on the assumption that the samples are "average", and will not require additional time beyond normal operations. Additional time must be allocated for samples which are very dirty or are extraordinary.

8. **REFERENCES** - The following USEPA methods are the official methods on which this Laucks Testing Laboratory method is based. The primary methods are those which most closely parallel the Laucks procedure and are referenced by their USEPA series and number. In those instances for which there are no official EPA methods, the most suitable reference is given under the miscellaneous references section. The additional reference section cites those methods which contain additional information. These methods will frequently be official methods, which apply in part to, or support the Laucks method.

PRIMARY REFERENCES:

Test Methods for Evaluating Solid Waste, USEPA, SW-846, Method 8330, (1994).

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-4002

Title: **Electronic Sample Entry and Log-In**

Revision history:

Number	Date
5	06/19/96
4	12/28/95
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Revised by: Pam Johnson, Sample Control Date: 6/19/96

Approved by: Karen Kotz, Laboratory Director Date: 6/20/96

Approved by: Harry Romberg, QC Officer Date: 6-19-96

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1. INTRODUCTION AND SCOPE

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.

1.2. 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.

1.3. 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
Waterproof labeling gun
PC work station linked to SAM

3. SAFETY PRECAUTIONS

3.1. 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 Inorganics) to perform operations when necessary (strong odors present, etc.), and wearing a respirator or dust mask if fumes or dust are generated.

3.2. 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.

3.3. 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 enterer from contamination may transfer contamination to these objects. Other persons may touch the

door knob or phone without glove protection and have the contamination transferred to their unprotected hands. Never put pens, paper clips, etc. in your mouth.

3.4. 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 air freight and overnight delivery services, and by Greyhound. It is the responsibility of the sample enterer to ensure that samples received by any of these services are promptly logged in and work requests made to the laboratory.

4.1.2. If a chain-of-custody (COC) is received with the sample set, sign it and record the date and time it was received. If the client has delivered the samples by hand, verify the cooler contents and return a copy of the COC to the client.

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 COC and the actual samples received are immediately reported to the client and are noted on the Sample Receipt Log. CLP Sample Receipt Log (Appendix 3) is for CLP log-in procedure. In-House Sample Receipt Log (Appendix 2) is for Laucks in-house 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. If there is a question about the integrity of the custody seals, make a note on the CLP Sample Receipt Log (Appendix 3); the client must be informed.

4.1.6. After the coolers are opened, determine whether there are soil or water samples in the coolers. Typically there will be a number of sample bottles for each sample if they are water; 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.

4.1.8. For any program (such as HAZWRAP, NFESC, or Army Corps) or other project-related samples 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 3). If any samples exceed the range of 4°C ± 2°C, the client must be contacted. In most cases, this should be done **in writing** (preferably FAX) by the appropriate project manager. A copy of the communication from the Supplemental Sample Receipt Log must be kept with the COC in the work order file.

4.1.9. Remove all bottles from the cooler and put on the bench. 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

4.1.10. For samples consisting of multiple containers, place all containers together on the bench. After all samples are arranged then check again Sample IDs, dates and times on each sample container.

4.1.11. 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 CLP Sample Receipt Log Form (Appendix 3).

4.1.12. All preserved water sample bottles for project-related work as well as unpreserved water sample bottles for HAZWRAP, NFESC, or Army Corps projects must also be checked for pH at the time of sample receipt. This is done by pouring out some of the sample into a small plastic cup and then using pH paper to record the pH at time of receipt. 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 3). If any samples exceed the pH requirements, the client must be contacted. In most cases, this should be done **in writing** (preferably FAX) by the appropriate project manager. The samples with inappropriate pH are listed on Laucks Testing Lab pH log form (Appendix 5) for corrective action. After the corrected preservation is completed this form is given to the appropriate project manager for work order filing.

4.1.13. Some samples are received at the lab that need to be split and preserved for different analytes. To accommodate preservation requirements, these samples are recorded on the

"Sample Split Sheet" (see Appendix 6). There is a specific cart located in sample entry where the samples are temporarily stored until splitting and preservation take place.

4.1.14. All sample container marks are then verified with each other and with the COC. This is done 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 In-House Sample Receipt Log (Appendix 2) or the CLP Sample Receipt Log (Appendix 3) and reported to the client.

4.1.15. To determine if the sample(s) is(are) acceptable, compare the existing conditions with the criteria specified in Appendix 7, "Required Containers/Volumes, Preservation Techniques and Maximum Holding Times for Environmental Analysis". All listed criteria must be met in order to qualify the sample(s) as "acceptable". If there are any problems with the sample(s) these must be documented in the "CLP Sample Receipt Log" (see Appendix 3). If any samples are not acceptable, the client must be contacted. In most cases, this should be done **in writing** (preferably FAX) 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 <u>if</u> that number applies to <u>all</u> 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 work order is started for the job through the ORD program. The work order is identified by a unique 7-digit number which is assigned by SAM at the time the work order 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 work order's sequence within the month. For instance, work order 9004001 was initiated in April, 1990 and was the first work order for that month.) This number will be used throughout the laboratory to track the job.

```
Laucks Testing        ORDER INFORMATION        01/13/94 11:42:21
9401492                                                              SAMPS
 KEYS    ST   ORDER # 94-01-496                RECEIVED 01/12/94      2
9401485 WR                                     DATE DUE 01/26/94
9401486 TR    CLIENT  H.H. NIECE        INVOICE BY R   VER BY
9401487 TR   PROJECT                     % DISCOUNT    KEEP FOR  45
9401488 WR   CONTACT KARIS               TEST/JOB      KEEP TIL 03/12/94   DP/TST
9401489 TR       CAT 1436759             % SURCHARGE       DISP D
9401490 TR                               #REP/INV  3
9401491 WR                                             QUOTED $            MS   4
9401492 WR   COMPANY H.H. Niece & Co.                  SAMPLE $ UNKNOWN
9401493 TR     FACIL Vashon, WA 98070                    MISC $
9401494 WR                                             TOTAL $ UNKNOWN
9401495 TR       REP Larry Niece                         INV #
9401496 WR     PHONE (206)463-5281
9401497 WR                                             CREATED 01/13/94
9401498 TR   WORK ID Wesleyan Community Church         WRITTEN 01/13/94
9401499 WR     TAKEN Client                          TRANSMIT
9401500 WR     TRANS UPS                             COMPLETE
9401501 WR      TYPE Water                           REPORTED
9401502 WR     ATTEN Larry Niece                     INVOICED
                P.O. #                            WRITTEN BY PAMJ
                                                                   CL NE SL J1
F1 | F2 | F3 | F4 | F5 | F6 | F7 | F8 | F9 | F10 |                  HELP
```

ORD Screen

4.2.3. The work order is to be filled out as completely as possible at this time. Above is an example of what the work order screen looks like on your PC. Typical information put into the work order screen (analogous to a cover page) includes:

- date of sample receipt;
- work order 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.

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.

4.2.4. All sample IDs, dates of collection, and dates of receipt are recorded in the FRAC program for the work order 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 and the project manager will notify the client.

```
Laucks Testing  FRACTION DESCRIPTION 04/05/94 13:36:36
9404T
  KEYS                                                  DEPTS/TESTS
9404176-12A   ORDER# 94-04-191/WR CON MARKB
9404176-13A   CLIENT PETER PAN    CAT                                  HIST1
              WRITTEN 04/05/94     BY HOLLYW                           HIST2
9404176-13B
9404176-13C     DASH # 02 A CATEGORY
9404176-13D  SAMPLE ID Mabi Mahi Lot #X3159 (#2)
9404176-14A  COLLECTED 04/05/94          QA/QC    MAT E
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

4.2.5. All sample bottles are numbered with the work order 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 required by Navy projects.

4.2.6. A sample can be uniquely identified by its work order number and the fraction number. For instance, if the work order number was 9004001 and there were 4 samples, the job would consist of samples

9004001-01
9004001-02
9004001-03
9004001-04

4.2.7. Each sample might consist of several containers, all of which would be marked with the same sample number.

4.2.8. 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 9004001-01 had 3 fractions, purely for internal accounting reasons, the three fractions would be identified as:

9004001-01A
9004001-01B
9004001-01C

4.2.9. 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 9004001-01.

4.2.10. For Navy projects, each bottle must have a unique bottle identifier. 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 bottle number 1.

4.2.11. 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.

4.2.12. ALERT: Each SAM work order can accommodate up to 57 fractions and no more. Each work order must allow sufficient fraction space for later changes or additions. Therefore, no more than 50 samples should be logged into any single work order. If, for administrative reasons, some or all of the samples consist of more than one fraction, then no more than 50 <u>fractions</u> can be logged. Should the submittal consist of more than 50 samples, or more than 50 fractions, initiate additional work orders as required. Cross-reference the work order 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 [Work Order Number]) or you can make appropriate comments in the Work Order Comment field (F2). To the degree possible, make sure that multiple work orders which represent one complete project in the client's mind are created sequentially, with no other unrelated work order numbers intervening.

4.2.13. Additionally, Sample Delivery Groups (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.

4.2.14. ALERT: Each fraction will accommodate only 27 tests. If more than 27 analyses are required on any sample, additional fractions should be made (ie -1A, -1B, -1C, etc.).

4.2.15. ALERT: The work order 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 work order, 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 documentation procedures for CLP samples

4.3.1. Completion of the CLP Sample Receipt Log, and the Supplemental Sample Receipt Log

4.3.1.1. 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 In-House Sample Receipt Log (Appendix 2). Copies of these forms may be found in Appendix 3.

4.3.1.2. 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
- work order #
- client project
- airbill number (if available)
- and initials of the person logging in the samples.

4.3.1.3. Complete the Non-Conformance check list. 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 work order file.

4.3.1.3.1. 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 is outlined in the Chain-Of-Custody SOP located in the SOP manual.

4.3.2. Assignment of SDG numbers

4.3.2.1. The SDG name is assigned by sample control and is usually based on client name or project name followed by sequential numbering.

4.3.3. Assignment of lab quality control samples

4.3.3.1. The client may choose to designate which samples are to be analyzed as matrix spike/matrix spike duplicate samples. 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.

4.3.3.1. Note in the **SAM SDG** records which sample is QC-assigned. This information is pulled into SDG from the FRAC program when the SDG is created.

4.3.3.2. 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.

4.3.3.3. Writing a department comments message for whichever department (EX, MS, and/or GC) should be notified.

4.3.4. Completion of SDG records.

4.3.4.1. For each project (NFESC, HAZWRAP, or Army Corps) 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.

4.3.4.2. 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 SPUMITO                           Updated:  02/19/93
BOGD5G   Project   : CDM (EDB)            Client:
BOGD5I   SAS Number:            Case Number:          Max. Samps:
BOGD6G
BOGD6I    Work  Samp                                                Associated
BOGD7G    Ord   Num  QC              Client ID          Mat TS pH    Blank
BOGD7I
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 SPUMITO                                     Updated:  02/19/93
BOGD5G              Project    : CDM (EDB)                     Client:
BOGD5I              SAS Number:                     Case Number:          Max. Samps:
BOGD6G
BOGD6I              Work  Samp                 Date
BOGD7G              Ord   Num    VTSR          Collected
BOGD7I
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  NU  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 SPUMITO                                     Updated:  02/19/93
BOGD5G              Project    : CDM (EDB)                     Client:
BOGD5I              SAS Number:                     Case Number:          Max. Samps:
BOGD6G                                                   Fractions
BOGD6I              Work  Samp
BOGD7G              Ord   Num    EDB
BOGD7I
BRPTSS
CANONS              9302598 17
CB2SFV              9302598 20
CB3-3V
CBSFXS
CDM10E
CDM10G
CDM10I
CDM10P
CDM10S
CDM10V
CDM11E
CDM11G                                                                  CL  NU  SL  J1
5=Lookup,Copy,Print  6=Toggle      F10=More    Shft-Fn=Fn Help
```

SDG, Screen 3

```
                                   SDG Database
CDM[redacted]   SDG Group : CDM10[redacted]     Date Due: 03/13/93[redacted]      Created:  02/19/93
  KEYS          Fraction  : E SPVMITO                                            Updated:  02/19/93
BOG05G          Project   : CDM CEDB[redacted]          Client: [redacted]
BOG05I          SAS Number: [redacted]      Case Number: [redacted]      Max. Samps: [redacted]
BOG06G
BOG06I
BOG07G
BOG07I
BRPTSS
CANONS
CB2SFU
CB3-3U
CBSFXS          Comments:
CDM10E[redacted]
CDM10G                [redacted]
CDM10I
CDM10P
CDM10S
CDM10U
CDM11E
CDM11G
[redacted]                                                                  CL NC SL J1
5=Lookup,Copy,Print  6=Toggle     F10=More    Shft-Fn=Fn Help
```

SDG, Screen 4

4.3.4.3. 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.

4.3.4.4. When the work order 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 work order number is entered, it is only necessary to enter sample numbers for subsequent samples from the same work order.)

4.3.4.5. 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.

4.3.4.6. 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.

4.3.4.7. The last screen page is used for any comments which the sample login person or project manager would like to record for the operations staff.

4.4. Sample storage

4.4.1. 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

4.4.2. 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.

4.4.3. For microbiological samples and for samples which arrive late in the day and for which the holding time will expire if the analysis is not started that day, the containers must be taken immediately 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.

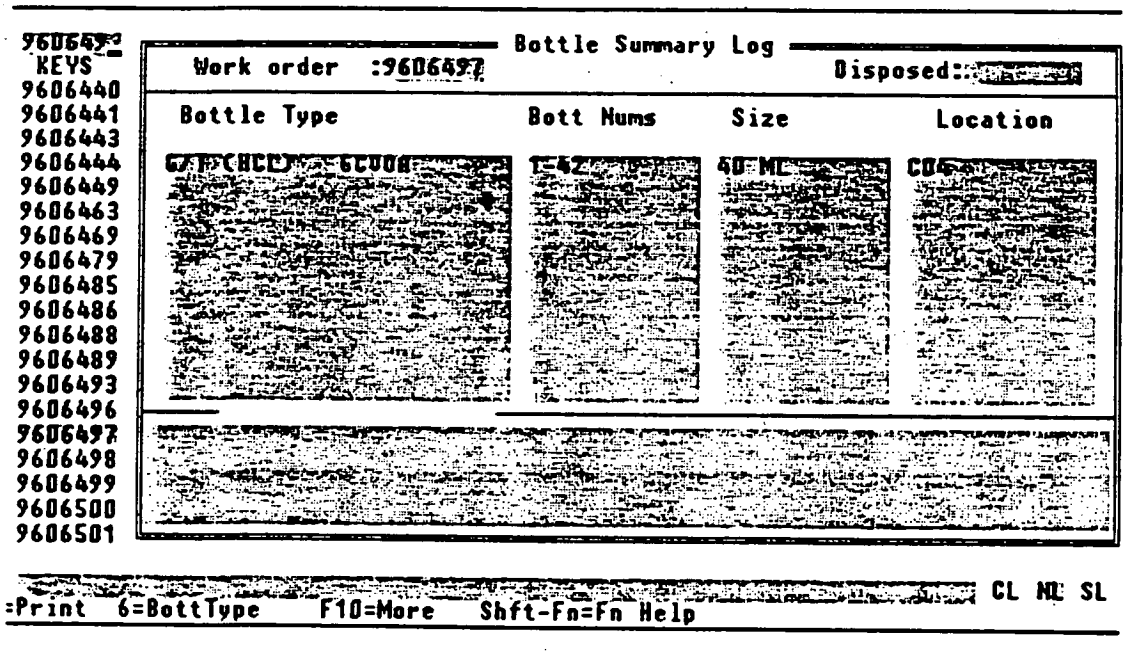
4.4.4. Storage locations

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.

4.4.4.1. *See Section 4.7 for transfer of extractable aliquots to the extractions lab

4.4.4.2. Prior to putting bottles into any storage location, the electronic Bottle Summary Log must be completed. For Navy projects, unique bottle identifiers must also be entered in the bottle log.

4.4.4.3. 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.



4.5. Determination of tests

4.5.1. 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.

4.5.2. 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.

4.5.3. Some soil samples will need to be shared between two or more departments. In order of priority, the following areas will receive samples in this order:

- If volatiles are requested, then the VOA departments will get the samples first (GC or GC/MS)
- The sample/samples will then be sent to the extractions lab
- The extractions lab will return the sample/samples to the inorganics lab or other areas

At the time of sample log-in the Sample Custodian will make appropriate comments for the department to return the samples to other departments for further testing. **Before** any samples are sent to other departments for testing, it is imperative that any requiring the analysis of volatile organics gets the sample first. Such samples should be given to the Volatiles Department before any other department.

4.6. Electronic Transmittal of Sample and Test Request Records

4.6.1. 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.

4.6.2. 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 work order and the sample fractions is clipped together by the Sample Custodian when sample log-in is complete. The work order number is written on, at a

minimum, the chain-of-custody document and may also be written on any other relevant documents.

4.6.3. 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 work order 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.

4.6.4. In performing the transmittal, it is the responsibility of the Project Manager, or designee, to double-check the work order 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.

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.

4.6.5. 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.

4.7. Generation of internal Chain-of-Custody (COC)

4.7.1. Samples which must be removed from the main building at 940 and taken to the extractions lab at 921 for preparation are tracked with an internal COC. This form is initiated by the person logging in the samples. The lab number, the client name, the number of samples, the sample matrixes and the enterer's initials and the date and time the form is started are recorded. See Appendix 4 for an example of an internal COC.

4.7.2. 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 log book at 921 before being placed in the cooler.

4.7.3. The COC is returned to 940 with the extracts when extractions are completed.

4.8. Sample breakage

4.8.1. All sample breakage, whether in shipping or while handling in the lab, must be reported immediately to the Project Manager.

4.8.2. If the sample was water, clean thoroughly with disposable towels. Be very careful with broken glass so as to avoid cuts.

4.8.3. 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.

4.8.4. All dirty, disposable clean up materials, soil, broken glass, etc. are placed in a plastic garbage bag before being placed in the dumpster. Any non-disposable clean up materials are washed after use.

4.9. Special circumstances

4.9.1. Samples from some clients are logged into a monthly work order. Some jobs extending over more than one sampling event may be entered under one work order number. In that event, pay special attention to date of receipt (see ALERT, above).

4.9.2. Other special circumstances may arise. If there are any questions, check with the Project Manager first.

APPENDIX 1

Sample Receipt Verification Stamp

Sample Receipt acknowledged pending verification of sample count. You will be notified within one working day of any discrepancies found

Signed _____ Date _____ Time _____

APPENDIX 2

In-House Sample Receipt Log

Laucks Testing Laboratories, Inc.
IN-HOUSE SAMPLE RECEIPT LOG

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 3

CLP Sample Receipt Log
Supplemental Sample Receipt Log

Laucks Testing Laboratories, Inc.
SAMPLE RECEIPT LOG (1) CLP

P:\PM\Holly\docs\fax\memo.doc12.22.94

Initial once samples are checked in _____

DATE RECEIVED: _____
TIME RECEIVED: _____
CLIENT NAME: _____
SDG # _____
COC # _____

SAMPLE LOG-IN DATE: _____
WORKORDER #: _____
CLIENT PROJECT: _____
AIRBILL ATTACHED?:(#) _____
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., Supplemental Sample Receipt Log

Work Order 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 4

Secure Storage Custody Log
Organic Extractions Custody Log

Laucks Testing Laboratories, Inc.

Secure Storage Custody Log

oject: _____ 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 Lab pH Log Form

Date_____

Analyst	WO Number	Fractions	pH before	Volume Acid Added/Type	Resulting pH

PHFORM.DOC

APPENDIX 6

Laucks Testing Lab Sample Split Sheet

Laucks Testing Lab Sample Split Sheet

WO #	FRAC #	WE HAVE	WE NEED	TOTAL	DISS	UNPRES	INTL & DATE	BOTTLE TYPE
								Y = YELLOW
								B = BLUE
								C = CYANIDE
								R = RED
								O = O&G
								P = PHENOL
								S = SULFIDE
								T = TOC
								OTHER =

APPENDIX 7

Required Containers/Volumes/Preservation/Holding Times

Laucks Testing Laboratories, Inc.

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 ≤ 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

Laucks Testing Laboratories, Inc.

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
Halogenated Volatile Organics	2 - 40 ml containers for liquids 20 grams for solids	Glass, Teflon-lined Septum, 40 ml capacity Above or Glass, 2-4 oz. capacity	Cool, 4° C, no headspace Cool, 4° C, packed to avoid headspace	14 days 14 days	Method 8010 GC/Hall - Direct Injection or Headspace, Method 5020 Purge-and-Trap, Method 5030 or Method 8260/Method 8240, GC/MS Purge-and-Trap Method 5030
Nonhalogenated Volatile Organics	2 - 40 ml containers for liquids 20 grams for solids	Glass, Teflon-lined Septum, 40 ml capacity Above or Glass, 2-4 oz. capacity	Cool, 4° C, adjust pH ≤ 2 with HCl, no headspace Cool, 4° C, packed to avoid headspace	14 days, with preservation 7 days, if not preserved 14 days	Method 8015 GC/FID Direct Injection or Headspace, Method 5020 Purge-and-Trap, Method 5030 or Method 8260/Method 8240, GC/MS Purge-and-Trap Method 5030

Laucks Testing Laboratories, Inc.

B. WASTE EVALUATION - ORGANICS ANALYSIS (SW-846, 3rd Edition) (continued)

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Aromatic Volatile Organics	2 - 40 ml containers for liquids 20 grams for solids	Glass, Teflon-lined Septum, 40 ml capacity Above or Glass, 2-4 oz. capacity	Cool, 4° C, if there is presence of residual chlorine then preserve with 0.5 g ascorbic acid and adjust pH to ≤2 with HCl, no headspace Cool, 4° C	14 days with preservation 14 days	Method 8020, GC/PID Direct Injection or Headspace, Method 5020 - Purge-and-Trap, Method 5030 Method 8260/8240, GC/MS Purge-and-Trap Method
Acrolein, Acrylonitrile Acetonitrile	2 - 40 ml containers for liquids 20 grams for solids	Glass, Teflon-lined Septum, 40 ml capacity Above or Glass, 2-4 oz. capacity	Cool, 4° C, adjust pH 4-5 with HCl, no headspace Cool 4° C, no headspace	14 days, with preservation 14 days	Method 8030, GC/FID Direct Injection or Headspace, Method 5020 - Purge-and-Trap Method 5030 - Groundwater using Method 5030 only. Method 8260/8240, GC/MS Purge-and-Trap Method
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 8080 GC/ECD

Laucks Testing Laboratories, Inc.

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 8140 GC/NPD or GC/NPD/ECD
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 8150 Extraction and Esterification/GC-ECD

Laucks Testing Laboratories, Inc.

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 20 grams for solids	Glass, Teflon-lined Septum 40 ml capacity As above or glass, 2-4 oz.	Cool, 4° C, acid preserved with HCl to pH < 2, no headspace Cool, 4° C, no headspace or if solid packed to minimize headspace	14 days 14 days	Method 8260 Purge-and-Trap GC/MS
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	----

Laucks Testing Laboratories, Inc.

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 Mercury, Total	100-200 ml for liquid sample Approximately 5 grams for solid sample	Plastic or Glass	HNO_3 to pH <2 for total Filter on site, HNO_3 to pH <2 for dissolved	28 days	EPA 245.1 for water or EPA 7470A 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.

Laucks Testing Laboratories, Inc.

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.

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D. METALS ANALYSIS (EPA Methods for Chemical Analysis of Water and Wastes, March 1983, or APHA Standard Methods, 15 Edition. and EPA SW-846, 3rd Edition) (continued)

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
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.

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D. METALS ANALYSIS (EPA Methods for Chemical Analysis of Water and Wastes, March 1983, or APHA Standard Methods, 15 Edition. and EPA SW-846, 3rd Edition) (continued)

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
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.

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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.

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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

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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

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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

Laucks Testing Laboratories, Inc.

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° 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° C, Methanol preservation may be a requirement of some states so state regulations must be consulted Cool, 4° 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° 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

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

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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
Fraction : G SPVMITO Updated: 10/23
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	Associate Blank
9510423	01		B952/SB-952-1/SS4,6-8/LK	S	88		B1018GSVS
9510423	02		B952/SB-952-1/SS9,16-18/LK	S	89		B1018GSVS
9510423	03		B952/SB-952-2/SS4,6-8/LK	S	83		B1018GSVS
9510423	04		B952/SB-952-2/SS5,8-10/LK	S	84		B1018GSVS
9510434	01		B952/SB-952-10/SS8,14-16LK	S	84		B1018GSVS
9510434	02		B952/SB-952-3/SS3,4-6/LK	S	84		B1018GSVS
9510434	03	MS	B 52/SB-952-3/SS6,10-12/LK	S	89		B1018GSVS
9510434	05	BMS	CH-5092500-S-SPOON-ERB-1	W			B1017GSVW
9510434	06		CH-POT-FB-1	W			B1017GSVW
9510434	07		CH-ASTM-5092500-FB-1	W			B1017GSVW
9510459	01		B952/SB-952-4/SS4,6-8/LK	S	84		B1018GSVS
9510459	02		B952/SB-952-4/SS7,12-14/LK	S	89		B1018GSVS
9510459	03		B952/SB-952-5/SS4,6-8/LK	S	87		B1018GSVS
9510459	04		B952/SB-952-5/SS6,10-12/LK	S	87		B1018GSVS
9510459	08		B952/SB-952-6/SS4,6-8/LK	S	84		B1018GSVS
9510459	09		B952/SB-952-6/SS6,10-12/LK	S	90		B1018GSVS
9510497	01		B952/SB-952-7/SS3,4-6/LK	S	89		B1018GSVS
9510497	02		B952/SB-952-7/SS8/4-16/LK	S	91		B1018GSVS
9510540	01		B952/SB-952-8/SS3,4.0-6.0	S	90		B1018
9510540	02		B952/SB-952-8/SS6,10.0-12	S	90		B1018GSVS

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	JP4	Fractions									
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:

SDG Database

SDG Group : GSI01 Date Due: 11/10/95 Created: 10/12/9
Fraction : I SPVMITO Updated: 10/17/9
Project : AFCEE/Chanute 952 Client: GSI (Inorganic)
PROJQC ID : GS_CHANUTE Max. Samps: 21

KEYS
080
08P
GLS08S
GLS08T
GLS08V
GLS09E
GLS09I
GLS09O
GLS09P
GLS09S
GLS09T
GLS09V
GSI01G
GSI01I
GSI01P
GSI01V
GSI02G
GSI02I
GSI02P

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

Appendix II

Example of Projqc in LIMS

Project QC Requirements Database

OHM
KEYS
HC_MANANN
:_MANANNS
nC_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

Proj Name: OHM-HAWAII Client Code: OHM_HAWAII Created: 12/19
Desc: UST sites Updated: 01/22
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,Ke

Project QC Requirements Database

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

Proj Name: OHM-HAWAII Client Code: OHM_HAWAII Created: 12/19
Desc: UST sites Updated: 01/22
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 Naphthalene, Acenaphthene, 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,BO

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,BO

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)
960312	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: _____

CHAIN OF CUSTODY RECORD: 4AP1313

Latich's Testing Laboratories, Inc.

WORK ORDER ID: 83[illegible]-2153 PAGE 1 OF 1

DATE 1-8-96

SUBMITTED AT: [illegible]

THIS INFORMATION TO BE USED FOR REPORTING/BILLING (SEE BELOW)

NAME: Roundwater Technology GSI

ADDRESS: 19033 W Valley Hwy D-101
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): [signature] (PRINTED NAME) Daryl Braun

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% SURC)
- [x] 5 DAYS (50% SURC)
- [ ] STD. 10-11 DAYS
- [ ] OTHER

TOTAL NO. OF CONTAINERS

CHAIN OF CUSTODY SEALS? [ ] YES [ ] NO [ ] NA

SHIPPED VIA [ ] UPS [ ] FED EX [ ] BUS [ ] HAND [ ] _____

TEMPERATURE _____

RELINQUISHED BY (SIGN AND PRINT)	DATE / TIME	RECEIVED BY (SIGN AND PRINT)	DATE / TIME
[signature] / Daryl Braun	1-8-96 1600	Pony Express	
		DAN D. JOHNSON	1/9/96

Laucks Testing Laboratories, Inc.
SAMPLE RECEIPT LOG (1) CLP

P:\PM\Holly\[illegible]

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: Danger Site A
AIRBILL ATTACHED?:(#) 2576393
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								

llowable temperature and pH ranges (neutral pH defined as a value between 5 and 9)

mperature	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

TMPLOG.DOT

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-4201

Title: **Package Deliverables for all Reporting Levels**

Revision history:

Number	Date
000	1/31/96

Written by: Tom Marino Date: 1-31-96

Reviewed by: Charlene Nix Date: 1-31-96

Reviewed by: Harry Romberg, QC Officer Date: 1-31-96

Approved by: Karen Kotz, Laboratory Director Date: 1/31/96

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 4
   2.1 Cover Page 4
   2.2 Narrative 5
   2.3 Chain-of-Custody Copies: 5
   2.4 Index: 5
   2.5 Organic fractions 8
   2.6 Inorganics Fractions: 10
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

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-Custody	Y	Y	Y	Y
Method Reference	Y	Y	Y	Y
Results	Y	Y	Y	Y
Method Blank		Y	Y	Y
SRM/LCS/BS		Y	Y	Y
MS/Dupe		Y		Y
MS/MSD			Y	Y
ICV			Y	Y
ICB			Y	Y
CCV			Y	Y
CCB			Y	Y
CRA			Y	Y
CRI			Y	Y
Post Spike			Y	Y
GFAA MSA			Y	Y
ICP Ser. Dil'n.			Y	Y
IDLs				Y
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
2	04/09/97
1	01/05/88

Written by: Bill Lundberg, Inorganics Supervisor Date: 4/9/97

Reviewed by: Harry Romberg, QA Officer Date: 4-9-97

Approved by: Karen Kotz, Laboratory Director Date: 4/9/97

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1. Introduction and Scope

1.1 Method Description

1.1.1 The purpose of this SOP is to define the procedures used in the inorganics department for the cleaning of glassware. The objective is to define a specific method of cleaning that is adapted to both the substances that are to be removed, and the determination to be performed.

1.2 Definitions

1.2.1 DIW = deionized water

2. Equipment List and Standards

2.1 Standard laboratory glassware, including but not limited to:

2.1.1 Volumetric flasks
Beakers
Funnels
Separatory funnels
Kjeldahl flasks
Nessler tubes
Erlenmeyer flasks
Burets
BOD bottles
Distillation Apparatus

3. Safety precautions and Waste Disposal

3.1 Safety Precautions

3.1.1 Several cleaning procedures or soaking procedures require the use of a chromic acid cleaning solution, 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. AGAIN, USE APPROPRIATE SAFETY PRECAUTIONS FOR SOLVENT USE! 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.

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.

4.1.2 ALKALINITY

4.1.2.1 Glassware Erlenmeyer flasks

4.1.2.2 Buret

4.1.2.3 Cleaning Procedures - Rinse with DIW.

4.1.2.4 Air dry.

4.1.3 AUTO ANALYZER (refer to the applicable analytical SOP)

4.1.4 BOD

4.1.4.1 Glassware BOD bottles

4.1.4.2 Glass pipettes

4.1.4.3 Cleaning procedure - Wash with hot tap water and Alconox.

4.1.4.4 Rinse with hot tap water.

4.1.4.5 Rinse with DIW.

4.1.4.6 Air dry.

4.1.5 COD

4.1.5.1 Procedure for Glassware Condensers, Erlenmeyer flasks, Buret

4.1.5.2 Cleaning Procedure - Rinse well with DIW only.

4.1.5.3 Soaking Procedure - Acid soak flasks with C.O.D. acid for 10 minutes prior to use. Rinse well with DIW.

4.1.6 CYANIDE

4.1.6.1 Procedure for Glassware, Volumetric flasks, Distillation apparatus

4.1.6.2 Cleaning procedure for Flasks - DIW rinse only.

4.1.6.3 Distillation apparatus - occasional H_2SO_4 and DIW wash, but generally DIW rinsed only.

4.1.7 Hexavalent Chromium

4.1.7.1 Cleaning procedure - Wash with hot tap water and Alconox.

4.1.7.2 Rinse well with DIW.

4.1.7.3 Air dry.

4.1.8 FORMALDEHYDES

4.1.8.1 Procedure for GlasswareBeakers, Erlenmeyer flasks, Volumetric flasks, Test tubes

4.1.8.2 Cleaning procedures - ,Rinse well with DIW only.

4.1.8.3 Never contaminate glassware with HNO_3 .

4.1.9 HARDNESS

4.1.9.1 Procedure for Glassware, Erlenmeyer flasks, Buret

4.1.9.2 Cleaning procedure - Rinse with DIW only.

4.1.10 KJELDAHL NITROGEN-LOW LEVELS (TKN AND AMMONIA)

4.1.10.1 Procedure for Glassware, Kjeldahl distillation apparatus, Kjeldahl flasks, Erlenmeyer, flasks or beakers

4.1.10.2 Cleaning procedure for Kjeldahl flasks - pre-distill with DIW and NaOH, and do final DIW rinses.

4.1.10.3 Beakers or Erlenmeyer flasks- DIW rinse only.

4.1.11 OIL AND GREASE

4.1.11.1 Procedure for Glassware, Volumetric flasks. Beakers, Funnels, Separatory funnels, Soxhlet digestion apparatus

4.1.11.2 Cleaning procedure for Vol. flasks, funnels, soxhlet apparatus- freon rinse 3-4 times.

4.1.11.3 Cleaning procedure for Beakers, separatory funnels- hot tap water and Alconox, rinse well with hot tap water, and dry.

4.1.12 METALS (INCLUDING HYDRIDES AND MERCURY)

4.1.12.1 Procedure for Glassware, Volumetric flasks. Beakers, Erlenmeyer flasks, BOD bottles, Digestion caps or watchglasses

4.1.12.2 Cleaning procedure - Rinse well with DIW only.

4.1.12.3 OPTIONAL:

4.1.12.4 If oily or difficult to clean, wash with acetone, scrub with hot tap water and Alconox, and rinse well with DIW.

4.1.12.5 Concentrated acid wash and DIW rinsed.

4.1.12.6 Bake in muffle furnace at 550 degrees C for 1 hour and rinse well with DIW.

4.1.12.7 NOTE: Some metals glassware should not be cleaned with Alconox at all.

4.1.12.8 Soaking procedure - Acid soak all metals glassware in HNO_3 and DIW.

4.1.12.9 Fill glassware with DIW and add 4-8 mls HNO_3. Cover and store until next use.

4.1.13 PHENOL

4.1.13.1 Procedure for Glassware, Kjeldahl flasks, Erlenmeyer flasks

4.1.13.2 Cleaning procedure for Kjeldahl flasks - pre-distill with H_2SO_4 and catalyst, DIW rinse.

4.1.13.3 Cleaning procedure for Erlenmeyer flasks - acid H_2SO_4 wash and DIW rinse.

4.1.13.4 NOTE: Use only those Kjeldahl flasks designated for use in phenol distillations.

4.1.14 PHOSPHATE

4.1.14.1 Cleaning procedure for GlasswareBeakers

4.1.14.2 Acid wash with H_2SO_4 .

4.1.14.3 Rinse with DIW.

4.1.15 TOC (SOILS)

4.1.15.1 Glassware, TOC combustion boats

4.1.15.2 Cleaning procedure - Brush out last sample.

4.1.15.3 Incinerate boat in muffle furnace at 950 degrees C.

4.1.16 TOX

4.1.16.1 Cleaning procedure for Glassware, Volumetric flasks, Misc. TOX glass parts

4.1.16.2 Cleaning procedure - Soak in chromic acid solution.

4.1.16.3 Wash with hot tap water and alconox.

4.1.16.4 Rinse well with DIW.

4.1.16.5 Bake in muffle furnace at 400 degrees C.

4.1.16.6 Cool.

4.1.16.7 Store in glass teflon sealed container inside a dessicator.

5. References

5.1.1 See the applicable analytical or preparation SOP for specific cleaning issues and references.

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-7009

Title: **Microwave Assisted Acid Digestion of Aqueous Samples and Extracts Using SW846 Method 3015**

Revision history:

Number	Date
1.	04/20/95
2.	06/04/96

Written by: Lucas Hawkins — Date: 6/4/96
Lucas Hawkins, Chemist

Reviewed by: Mark J. Strongman — Date: 6.4.96
Mark Strongman, Metals Supervisor

Reviewed by: Bill Lundberg — Date: 6/4/96
Bill Lundberg, Inorganic Division Manager

Reviewed by: Harry Romberg — Date: 6-4-96
Harry Romberg, QC Officer

Approved by: Karen J. Kotz — Date: 6/4/96
Karen Kotz, Laboratory Director

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1. Introduction and Scope

1.1 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.

1.2 This digestion procedure is used for the preparation of aqueous samples, mobility-procedure extracts, and wastes that contain suspended solids for analysis by flame atomic absorption spectroscopy (FLAA), graphite furnace absorption spectroscopy (GFAA), inductively coupled argon plasma spectroscopy (ICP), or inductively coupled argon plasma mass spectrometry (ICP-MS). The procedure is a hot acid leach for determination of available metals.

1.3 Samples prepared by Method 3015 using nitric acid digestion may be analyzed by FLAA, GFAA, ICP or ICP-MS for the following:

Aluminum	Calcium	Lead	*Selenium
Antimony	Chromium	Magnesium	Silver
*Arsenic	Cobalt	Manganese	Sodium
Barium	Copper	Molybdenum	Thallium
Beryllium	Iron	Nickel	Vanadium
Cadmium	Zinc	Potassium	

*Cannot be analyzed by FLAA

1.4 Microwave power settings and times used in this SOP are based on manufacturers recommendation. Laucks system is different from the design used in both the SW846 and EPA CLP publications, in that it uses a 12 place carousel rather than 5 and that our system does not currently allow us to monitor the temperature in the vessels.

2. Summary of Method

2.1 Nitric acid is added to an aqueous sample in a 120 mL Teflon® digestion vessel. The vessel is capped and heated in a microwave unit. After cooling, the vessel contents are filtered, or allowed to settle in a clean sample bottle for analysis.

3. Safety Procedures

3.1 These procedures involve the use of strong and/or hot acid solutions. The analyst must wear eye protection, a lab coat, and gloves to protect against burns.

3.2 Samples and spiking solutions may contain high levels of toxic metals and other unknown constituents. The analyst must take every precaution to avoid contact with these

potentially hazardous materials and should wash hands thoroughly before eating or leaving the laboratory.

3.3 Very reactive or volatile materials may create high pressures when heated, causing the vessels to vent with potential loss of sample and analytes. Samples that contain carbonates or other carbon dioxide generating compounds may generate enough pressure to vent the vessel. If this situation is anticipated the analyst may wish to use a smaller sample or an alternative digestion method.

4. Equipment

- MDS-205 Microwave Digestion system, CEM
- Capping station
- Carousel
- 120 mL Teflon® vessels and caps
- 50 or 100 mL graduated cylinder, acid washed
- Glass funnels, acid-washed
- Filter paper, Whatman No. 41 or equivalent
- Sample digestate bottles, acid washed
- Analytical balance, 300 g capacity, minimum ± 0.01 g.

Note: All glassware used for this digestion must be prepared by the method stated in Inorganics Glassware Cleaning Procedures, LTL-7003.

4.1 All Teflon® vessels and caps are to be thoroughly rinsed with deionized water prior to digestions and shaken to remove any significant droplets in the vessels.

4.2 The microwave is to be calibrated by using the method stated in SW846 method 3051.

5. Reagents

5.1 Reagent Water - Reagent water will be interference free. All references to water in the method refer to reagent water unless otherwise specified.

5.2 Nitric acid (concentrated), HNO_3, ACS Reagent grade or better

The method blank, prepared along with the samples, is used as a contamination check. Since the holding time for all analytes associated with this method have a holding time of 6 months, repreparation would not be an issue if contamination was traced to a specific reagent.

6. Microwave Sample Digestion Procedure

See Appendix A for sample digestion log.
See Appendix B for flowchart.
See Appendix C for QC Solutions

6.1 A 45 mL aliquot of well shaken sample is measured into a graduated cylinder. This aliquot is poured into the Teflon® digestion vessel with the number of the vessel recorded on the preparation sheet.

6.2 A blank sample of reagent water is treated in the same manner along with spikes, duplicates, and laboratory control samples. If only one graduated cylinder is used for all samples, the preparation blank is to be the last sample dispensed to assure the absence of sample carry-over.

6.3 Add 5 mL of concentrated nitric acid to each vessel and cap hand tight. For each microwave digestion run, a double ported lid is to accompany the sample that seems to have the most carbon. Weigh each capped vessel to the nearest 0.01 g.

6.4 Place the carousel in the unit and attach the pressure control line to the digestion vessel with the double ported lid. Set the first program to run at 100% power for approximately 15 minutes., and the second program for 100% power for 10 minutes. Adjust the pressure control unit to 70 psi. After starting the microwave, monitor the pressure until it reaches the 70 psi limit. Advance the microwave to the second program and re-start.

6.5 At the end of the microwave program, allow the vessels to cool for at least 5 minutes in the unit before removal to avoid possible injury if a vessel vents immediately after microwave heating. Cool the samples before re-weighing to determine the extent of sample loss if any. If the weight of the sample plus acid has decreased by more than 10%, discard and prepare the sample again.

6.6 Uncap the vessels and transfer the samples to an acid-cleaned polyethylene bottle. If the digested sample contains particulate which may clog nebulizers or interfere with injection of the sample into an instrument, the sample may be allowed to settle or be filtered.

6.7 The prepared samples are then transferred to the metals analysis department. The digestion log is to reflect the time at the start and finish of the digest. In order to maintain a strict chain of custody, the time and date when the digestates are relinquished to the analysis department, as well as the initials of the analyst accepting the digestates, are recorded on the digest log

7. Quality Control

7.1 In each batch or SDG consisting of no more than 20 samples, a preparation blank is created. The blank should be labeled on the digestate bottle in the following way: B, date,

M (microwave), W (water) and the sequence number of the digestion. Example B022095MW01.

7.2 Spiked samples should be employed to determine accuracy. In each batch or SDG consisting of no more than 20 samples, either matrix spike / sample duplicate (MS/Dup) or matrix spike /matrix spike duplicate (MS/MSD) samples will be prepared. For SW3015-specified quality control measures, MS/Dup samples will be prepared in the following way. Dispense a sample in triplicate. Two will be analyzed exactly as any other sample. The third must be spiked with the appropriate spike solutions, determined by the analyst. 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 aliquot must be spiked.

7.3 In each batch or SDG consisting of no more than 20 samples, prepare a Lab Control Sample (LCSW) in exactly the same manner as a sample. Each spiking solution is given its own unique number according to the page and line of the standards logbook which it is entered into. This number and the volume dispensed must be clearly recorded on the digestion log. A sample page from the standards logbook is included in Appendix C.

8. References

8.1 USEPA, Test Methods for Evaluating Solid Waste Physical/Chemical Methods, USEPA SW846, most recent version, Method 3015.

Appendix A, Sample LTL-7009 Digestion Log

Laucks Testing Laboratories, INC.	START DATE TIME	FINISH DATE TIME	METHOD	ANALYST	ANALYSIS
SDG #:			SW846		Microwave
MATRIX: WATER			3015		Digestion

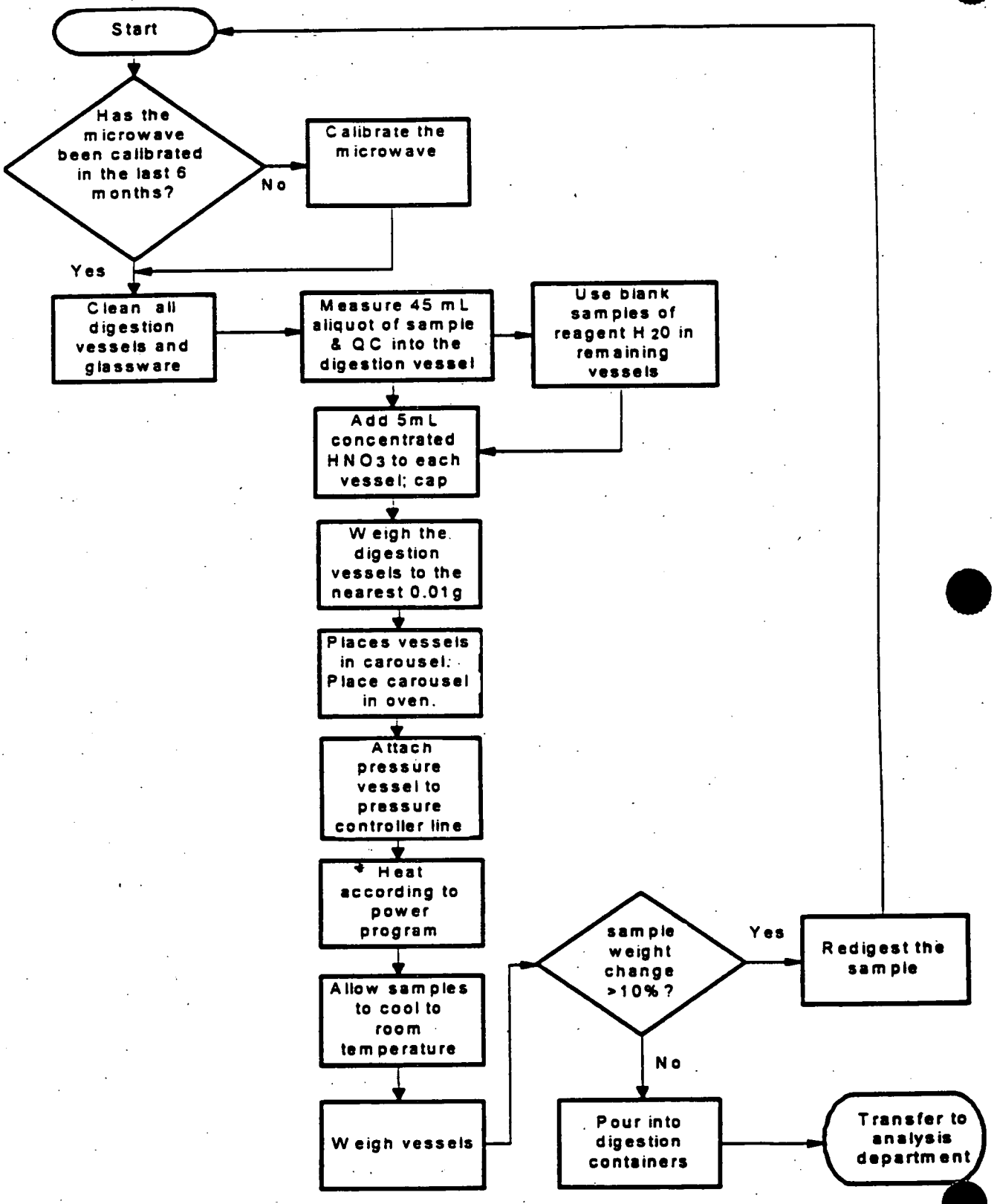
Bomb #	Sample ID	Samp Vol (mL)	Final Vol (mL)	Color		Clarity		Bomb & Sample (g)	Final Bomb (g)	Δ (g)	Δ >2.5 g ?
				Int	Fin	Int	Fin				

Chain of Custody	Digest Relinquished Date / Time : Analyst	Digest Accepted :Analyst	Digest Disposed :Date
	/ :		

COLOR: 1 = Red, 2 = Blue, 3 = Yellow, 4 = Green, 5 = Orange, 6 = Violet, 7 = White, 8 = Brown, 9 = Gray, 10 = Black, 11 = Colorless.

CLARITY: 1 = Clear, 2 = Cloudy, 3 = Opaque.

Appendix B, Flowchart for LTL-7009


Start
Has the microwave been calibrated in the last 6 months?
No
Calibrate the microwave
Yes
Clean all digestion vessels and glassware
Measure 45 mL aliquot of sample & QC into the digestion vessel
Use blank samples of reagent H 20 in remaining vessels
Add 5mL concentrated HNO3 to each vessel; cap
Weigh the digestion vessels to the nearest 0.01g
Places vessels in carousel: Place carousel in oven.
Attach pressure vessel to pressure controller line
Heat according to power program
Allow samples to cool to room temperature
Weigh vessels
sample weight change >10%?
Yes
Redigest the sample
No
Pour into digestion containers
Transfer to analysis department

Appendix C, Quality Control Solutions

ICP LCSW

The LCSW for the ICP digest was made by diluting 100 mLs of SPEX ICV-1™ solution to 1000 mLs with type II water. A 100 mL aliquot of this solution was then digested.

ICP Analytes

Analyte	Concentration in Digest (ppb)	Analyte	Concentration in Digest (ppb)
Al	20000	Pb	500
Ba	20000	Mg	500000
Be	500	Mn	1500
Cd	500	Ni	4000
Ca	500000	K	500000
Cr	1000	Ag	1000
Co	5000	Na	500000
Cu	2500	V	5000
Fe	10000	Zn	2000
Sb	6000		

Other Analytes

Analyte	Concentration in Digest (ppb)	Analyte	Concentration in Digest (ppb)
As	1000	Tl	1000
Se	500		

Tables based on a final volume of 100 mL.

Graphite Furnace LCSW

The LCSW for the Graphite Furnace digest was made by diluting 100 mLs of SPEX ICV-1™ solution to 1000 mLs with Type II water. A 10 mL aliquot of this solution was added to 90 mL of Type II water and then digested.

Graphite Furnace Analytes

Analyte	Concentration in Digest (ppb)	Analyte	Concentration in Digest (ppb)
As	100	Tl	100
Pb	50	Sb	600
Se	50		

Other Analytes

Analyte	Concentration in Digest (ppb)	Analyte	Concentration in Digest (ppb)
Al	2000	Pb	50
Ba	2000	Mg	50000
Be	50	Mn	150
Cd	50	Ni	400
Ca	50000	K	50000
Cr	100	Ag	100
Co	500	Na	50000
Cu	250	V	500
Fe	1000	Zn	200

Tables based on a final volume of 100 mL.

ICP Spikes

- The CLP spike solution for ICP digest was made by diluting 10 mLs of CLPP-SPK-1™, 10 mLs of CLPP-SPK-2™, and 10 mLs of CLPP-SPK-3™ to 100 mLs with Type II water.

- Add 1000 µLs of CLP spike solution to water samples and 2000 µLs of CLP spike solution to soil samples prior to digestion.

ICP Analytes

Analyte	Concentration in Digest (ppb)	Analyte	Concentration in Digest (ppb)
Ag	50	Fe	1000
Al	2000	Mn	500
As	2000	Ni	500
Ba	2000	Pb	500
Be	50	Se	2000
Cd	50	Sb	500
Co	500	Tl	2000
Cr	200	V	500
Cu	250	Zn	500

Table based on a final volume of 100 mL for water digests, and 200 mL for soil digests.

GRAPHITE FURNACE SPIKES

- Add the analytes listed in the table below to a 100 mL volumetric flask which contains ~50 mL of Type II water and 5 mL of HNO_3.

Analyte	Volume	Stock Concentration
As	400 µL	1000 ppm
Pb	200 µL	1000 ppm
Se	100 µL	1000 ppm
Tl	500 µL	1000 ppm

- Dilute to 100 mL with Type II water. This spike solution now contains:

4.0 ppm	Arsenic
2.0 ppm	Lead
1.0 ppm	Selenium
5.0 ppm	Thallium

- Add 1000 µL of Spike Solution to water samples and 2000 µL to soil samples prior to digestion.

ICP
CLP-LOW SPIKES

- Add the analytes from the two tables below into separate 100 mL volumetric flasks which contain ~50 mL of Type II water and 5 mL of HNO_3.

ICP-1

Analyte	Volume	Stock Concentration	Spike Concentration
Sb	500 µL	1000 ppm	5 ppm
Al	5000 µL	1000 ppm	50 ppm
Ba	2000 µL	1000 ppm	20 ppm
Be	100 µL	1000 ppm	1 ppm
Cr	500 µL	1000 ppm	5 ppm
Co	1000 µL	1000 ppm	10 ppm
Cu	500 µL	1000 ppm	5 ppm
Fe	2500 µL	1000 ppm	25 ppm
Mn	500 µL	1000 ppm	5 ppm
Ni	1000 µL	1000 ppm	10 ppm
V	1000 µL	1000 ppm	10 ppm
Zn	1000 µL	1000 ppm	10 ppm

ICP-2

Analyte	Volume	Stock Concentration	Spike Concentration
Ag	500 µL	1000 ppm	5 ppm

- Add 1000 µL of ICP-1 and 1000 µL of ICP-2 spike solutions to water samples prior to digestion. For soil samples, add 2000 µL of each spike solution prior to digestion. The final digest will contain:

Analyte	Concentration in Digest	Analyte	Concentration in Digest
Sb	50 ppb	Al	500 ppb
Ba	200 ppb	Be	10 ppb

Cr	50 ppb	Co	100 ppb
Cu	50 ppb	Fe	250 ppb
Mn	50 ppb	Ni	100 ppb
V	100 ppb	Zn	100 ppb
Ag	50 ppb		

Table based on final volume of 100 mL for water digests, and 200 mL for soil digests.

GRAPHITE FURNACE
CLP-LOW SPIKES

- Add the analytes listed in the table below to a 100 mL volumetric flask which contains ~50 mL of Type II water.

Analyte	Volume	Stock Concentration	Spike Concentration
As	100 µL	1000 ppm	1000 ppb
Se	500 µL	1000 ppm	5000 ppb
Tl	500 µL	1000 ppm	5000 ppb
Pb	250 µL	1000 ppm	2500 ppb
Cd	100 µL	1000 ppm	1000 ppb

- Add 1000 µL of Spike Solution to water samples and 2000 µL to soil samples prior to digestion. The final digest will contain*:

10 ppb Arsenic
50 ppb Selenium
50 ppb Thallium
25 ppb Lead
10 ppb Cadmium

*Based on 100 mL final volume for water digests and 200 mL for soil digests.

PDCA
SPIKES and LCSW

The LCSW for the PDCA extraction was make by adding 10 mL SPEX ICV-1™ solution to 100 mL of Type II water prior to extraction. The spike was made by adding 1000 µL of CLP SPK solution to a 100 mL aliquot of the sample prior to extraction.

ICP Analytes

Analyte	Concentration in Digest	Analyte	Concentration in Digest
Cd	50 ppb	Mn	500 ppb
Co	500 ppb	Ni	500 ppb
Cu	250 ppb	Ag	50 ppb
Fe	1000 ppb	V	500 ppb
Zn	500 ppb		

Graphite Furnace Analytes

Analyte	Concentration in Digest	Analyte	Concentration in Digest
Pb	500 ppb	Tl	2000 ppb
Se	2000 ppb		

Tables based on a final volume of 100 mL.

LAUCKS TESTING LABORATORIES, INC.
STANDARDS LOGBOOK

#	ANALYTE	ID or STOCK No.	STOCK CONC.	VOL/WT TAKEN	FINAL VOL.	CONC.	SOLVENT	PREP. DATE	INIT.	EXP. DATE	QC'D (Initials)
1	CLP.SPK	ME1.03.06 EXP 12/12/96	(CLPP SPK 1)	10mL	100mL		5% HNO_3 H_2O	03/28/96	LJH	12/12/96	
		ME1.03.07 EXP 12/12/96	(CLPP.SPK 2)								
		ME1.03.09 EXP 12/12/96	(CLPP.SPK 3)								
2	GF-CAL1 (Pb)	ME3.86.09 EXP. 7/10/96	1000 S	30µL	100mL	0.3 ppm	0.5% HNO_3 H_2O	03/28/96	LJH	05/30/96	
	(Se)	ME3.86.10 EXP 7/10/96		50µL		0.5 ppm					
	(As)	ME3.81.9 EXP 5/30/96		100µL		1.0 ppm					
	(Tl)	ME3.86.11 EXP 7/10/96		100µL		1.0 ppm					
3	GF-CAL2 (Pb)	ME3.86.09 EXP 7/10/96	1000 S	100µL	100mL	1.0 ppm	0.5% HNO_3 H_2O	03/28/96	LJH	05/30/96	
	(Se)	ME3.86.10 EXP 7/10/96		100µL		1.0 ppm					
	(As)	ME3.81.9 EXP 5/30/96		250µL		2.5 ppm					
	3/28/96 LJH										

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

Method #: LTL-7010

Title: **Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FLAA or ICP Spectroscopy by SW846 Method 3010A**

Revision history:

Number	Date
1.	12/14/94
2.	06/03/96

Written by: Lucas Hawkins, Chemist — Date: 6/4/96

Reviewed by: Mark Strongman, Metals Supervisor — Date: 6.3.96

Reviewed by: Bill Lundberg, Inorganic Division Manager — Date: 6/4/96

Reviewed by: Harry Romberg, QC Officer — Date: 6-4-96

Approved by: Karen Kotz, Laboratory Director — Date: 6/4/96

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1. Introduction and Scope

1.1 This digestion procedure is used for the preparation of aqueous samples, TCLP and mobility-procedure extracts, and wastes that contain suspended solids for analysis by flame atomic absorption spectroscopy (FLAA) or inductively coupled argon plasma spectroscopy (ICP). The procedure is used to determine total metals.

1.2 Samples prepared by Method 3010A may be analyzed by FLAA or ICP for the following:

Aluminum	Calcium	Magnesium	*Silver
*Antimony (also GFAA)	Chromium	Manganese	Sodium
**Arsenic	Cobalt	Molybdenum	Thallium
Barium	Copper	Nickel	*Tin (FLAA only)
Beryllium	Iron	Potassium	Vanadium
Cadmium	Lead	**Selenium	Zinc

* Although Antimony, Silver and Tin are not listed in the original SW846 document as analytes that may be analyzed from samples prepared by Method 3010A, Laucks has demonstrated adequate recovery for these analytes from this digestate. In addition, Laucks has demonstrated adequate Antimony recovery in samples prepared by LTL-7010 and analyzed by GFAA.

** Analysis by ICP

1.3 With the exception of Antimony, this digestion procedure is not suitable for samples which will be analyzed by graphite furnace atomic absorption spectroscopy because hydrochloric acid can cause interference during furnace atomization. Consult Method LTL-7008 for samples requiring graphite furnace analysis.

2. Summary of Method

2.1 A mixture of nitric acid and the material to be analyzed is refluxed in a covered beaker. This step is repeated with additional portions of nitric acid until the digestate is light in color or until its color has stabilized. After the digestate has been brought to a low volume, it is refluxed with hydrochloric acid and brought up to volume. If a sample goes to dryness, it must be discarded and prepared again.

3. Safety Procedures

3.1 These procedures involve the use of strong and/or hot acid solutions. The analyst must wear eye protection, a lab coat, and gloves to protect against burns.

3.2 These procedures involve hot plates which may present the danger of burns from heated surfaces and electrical hazards. The analyst must take appropriate caution to avoid injury from these sources.

3.3 Samples and spiking solutions may contain high levels of toxic metals and other unknown constituents. The analyst must take every precaution to avoid contact with these potentially hazardous materials and should wash hands thoroughly before eating or going home for the day.

4. Equipment

- 150-mL beakers or equivalent, acid-washed
- 100-mL graduated cylinder, acid-washed
- Ribbed watch glasses, acid-washed
- Eppendorf or other micropipets
- Hot plate or equivalent heating source, one that is adjustable and capable of maintaining a temperature of 90-95° C.
- Glass funnels, acid-washed
- Filter paper, Whatman No. 41 or equivalent
- 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, LTL-7003.

5. Reagents

5.1 Reagent Water. Reagent water will be interference-free. All references to water in the method refer to reagent water unless otherwise specified.

5.2 Nitric acid (concentrated), HNO_3, ACS Reagent grade or better.

5.3 Hydrochloric acid (concentrated), HCl, ACS Reagent grade or better.

The method blank, prepared along with the samples, is used as a contamination check. Since the holding time for all analytes associated with this method have a holding time of 6 months, repreparation would not be an issue if contamination was traced to a specific reagent.

6. Acid Digestion Procedure

See Appendix A for digestion log.
See Appendix B for flowchart.
See Appendix C for Quality Control solutions.

6.1 Transfer a 100-mL representative aliquot of the well-mixed sample to a 150-mL beaker and add 3 mL of concentrated HNO_3. If only one graduated cylinder is used for all samples, the preparation blank is to be the last sample dispensed to assure the absence of sample carry-over. Cover the beaker with a ribbed watch glass. Place the beaker on a hot plate and carefully evaporate to 5 mL, making certain that the sample does not boil and that no portion of the bottom of the beaker is allowed to go dry. Place a thermometer into a beaker of water on the hotplate. Record the temperature achieved during the digest on the digest log. Cool the beaker and add another 3-mL aliquot of HNO_3. Return the covered beaker to the hot plate and adjust the temperature, if necessary, so that a gentle reflux action occurs.

6.2 Continue heating, adding additional HNO_3 as necessary until the digestion is complete, indicated when the digestate is light in color or does not change in appearance upon additional heating. Evaporate the samples to 3 mL, not allowing any portion of the bottom of the beaker to go dry. After allowing the beakers to cool, add 5 mL of concentrated HCl and 5 mL reagent water. Reflux the samples for an additional 15 minutes to dissolve any precipitate or residue resulting from evaporation.

Note: If a sample is allowed to go to dryness at any stage of the digestion procedure, low recoveries will result. The sample must be discarded and prepared again.

6.3 After cooling the beakers, add 5mL concentrated HCl to obtain a final acid concentration of approximately 10%. Pour each sample into a 100-mL graduated cylinder, rinsing the beaker walls with water and adding the rinsate to the graduated cylinder, and bring to a final volume of 100 mL. If one graduated cylinder is to be used to adjust volume of each sample, the blank is to be the last sample dispensed. If silicates or other insoluble material that could clog the nebulizer are present in the samples, the samples should be allowed to settle overnight prior to analysis. Filtration should be performed only if there is concern that the insoluble material will not settle out of solution. Filter and dilute the samples to a final volume of 100 mL, using acid-washed filter apparatus to avoid sample contamination. Samples should be stored in acid-washed digestate bottles.

6.4 The prepared samples are then transferred to the metals analysis department. The digestion log is to reflect the time at the start and finish of the digest. In order to maintain strict chain of custody, the time and date when the digestates are relinquished to the analysis department, as well as the initials of the analyst accepting the digest, are recorded on the digestion log.

7. Quality Control

7.1 In each batch or SDG consisting of no more than 20 samples, a preparation blank is created. This consists of 100 mL of reagent water and the appropriate amounts of nitric and hydrochloric acid, digested in exactly the same way as the samples. If only one graduated cylinder is used for all samples, the preparation blank is to be the last sample

dispensed to assure the absence of sample carry-over. The blank should be labeled on the digestate bottle in the following way: B, date, instrument, S or W (for soil or water) and the sequence number of the digestion. Example: B111794ICPW01.

7.2 Spiked samples should be employed to determine accuracy. In each batch or SDG consisting of no more than 20 samples, either matrix spike / sample duplicate (MS/Dup) or matrix spike / matrix spike duplicate (MS/MSD) samples will be prepared. For SW846-specified quality control measures, MS/Dup samples will be prepared in the following way. Dispense one sample in triplicate. Two will be digested exactly as any other sample. The third must be spiked with the appropriate spike solutions determined by the analyst, and then treated as any other sample. All glassware and digestate bottles must be marked appropriately. If MS/MSD samples are required instead of the SW846-specified MS/Dup, both the second and third aliquots must be spiked and all glassware and digestate bottles marked appropriately.

7.3 Determine the analytes required for analysis by consulting sample work order information. Each spiking solution is given its own unique number according to the page and line of the standards logbook which it is entered into. This number and the volume dispensed must be clearly recorded on the digestion log. A sample page from the standards logbook is included in Appendix C.

7.4 In each batch or SDG consisting of no more than 20 samples, either a Blank Spike (BLK SPK) or a Laboratory Control Sample (ICP LCSW) must be digested in exactly the same manner as a sample. A Blank Spike consists of 100 mL of reagent water spiked at the levels determined by the analyst as outlined in Section 7.3.

8. References

8.1 USEPA, Test Methods for Evaluating Solid Waste Physical/Chemical Methods, USEPA SW846, most recent version, Method 3010A.

Appendix A, Sample LTL-7010 Digestion Logs

LAUCKS TESTING LABORATORIES, INC.			ICP DIGESTION LOG		Place a √ in the box next to the method performed.			
SDG #:	Digest start : date/time		Digest finish : date/time		SW-846 3010A		CLP-water	
Analyst:	/		/		SW-846 3050A		CLP-soil	

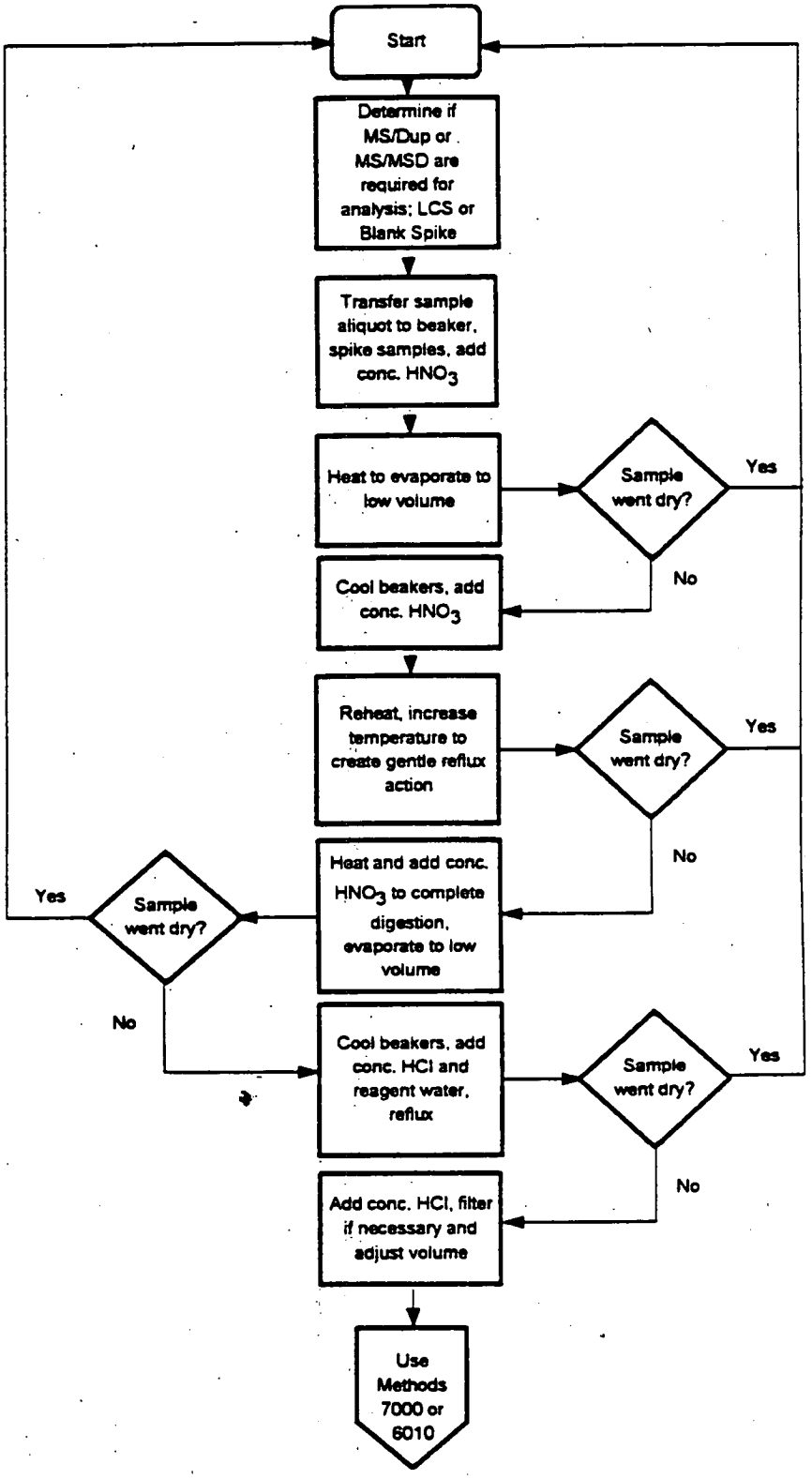
Sample ID	Sample Size (g:mL)	Final Volume (mL)	Color Bef	Color Aft	Clarity Bef	Clarity Aft	Text	Art	Comments

Chain of Custody	Digest Relinquished Date / Time : Analyst	Digest Accepted Analyst	Digest Disposed Date	Hot Plate Temperature, °C
	/			

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) Artifacts: If "Yes", give description in the comments field.

P:\METALS\BNCHSHEET\DIG.D

Appendix B, Flowchart for LTL-7010


Start
Determine if MS/Dup or MS/MSD are required for analysis; LCS or Blank Spike
Transfer sample aliquot to beaker, spike samples, add conc. HNO_3
Heat to evaporate to low volume
Sample went dry?
Yes
No
Cool beakers, add conc. HNO_3
Reheat, increase temperature to create gentle reflux action
Sample went dry?
Yes
No
Heat and add conc. HNO_3 to complete digestion, evaporate to low volume
Sample went dry?
Yes
No
Cool beakers, add conc. HCl and reagent water, reflux
Sample went dry?
Yes
No
Add conc. HCl, filter if necessary and adjust volume
Use Methods 7000 or 6010

Appendix C, Quality Control Solutions

ICP LCSW

The LCSW for the ICP digest was made by diluting 100 mLs of SPEX ICV-1™ solution to 1000 mLs with type II water. A 100 mL aliquot of this solution was then digested.

ICP Analytes

Analyte	Concentration in Digest (ppb)	Analyte	Concentration in Digest (ppb)
Al	20000	Pb	500
Ba	20000	Mg	500000
Be	500	Mn	1500
Cd	500	Ni	4000
Ca	500000	K	500000
Cr	1000	Ag	1000
Co	5000	Na	500000
Cu	2500	V	5000
Fe	10000	Zn	2000
Sb	6000		

Other Analytes

Analyte	Concentration in Digest (ppb)	Analyte	Concentration in Digest (ppb)
As	1000	Tl	1000
Se	500		

Tables based on a final volume of 100 mL.

ICP Spikes

- The CLP spike solution for ICP digest was made by diluting 10 mLs of CLPP-SPK-1™, 10 mLs of CLPP-SPK-2™, and 10 mLs of CLPP-SPK-3™ to 100 mLs with Type II water.

- Add 1000 µLs of CLP spike solution to water samples and 2000 µLs of CLP spike solution to soil samples prior to digestion.

ICP Analytes

Analyte	Concentration in Digest (ppb)	Analyte	Concentration in Digest (ppb)
Ag	50	Fe	1000
Al	2000	Mn	500
As	2000	Ni	500
Ba	2000	Pb	500
Be	50	Se	2000
Cd	50	Sb	500
Co	500	Tl	2000
Cr	200	V	500
Cu	250	Zn	500

Table based on a final volume of 100 mL for water digests, and 200 mL for soil digests.

ICP
CLP-LOW SPIKES

- Add the analytes from the two tables below into separate 100 mL volumetric flasks which contain ~50 mL of Type II water and 5 mL of HNO_3.

ICP-1

Analyte	Volume	Stock Concentration	Spike Concentration
Sb	500 µL	1000 ppm	5 ppm
Al	5000 µL	1000 ppm	50 ppm
Ba	2000 µL	1000 ppm	20 ppm
Be	100 µL	1000 ppm	1 ppm
Cr	500 µL	1000 ppm	5 ppm
Co	1000 µL	1000 ppm	10 ppm
Cu	500 µL	1000 ppm	5 ppm
Fe	2500 µL	1000 ppm	25 ppm
Mn	500 µL	1000 ppm	5 ppm
Ni	1000 µL	1000 ppm	10 ppm
V	1000 µL	1000 ppm	10 ppm
Zn	1000 µL	1000 ppm	10 ppm

ICP-2

Analyte	Volume	Stock Concentration	Spike Concentration
Ag	500 µL	1000 ppm	5 ppm

- Add 1000 µL of ICP-1 and 1000 µL of ICP-2 spike solutions to water samples prior to digestion. For soil samples, add 2000 µL of each spike solution prior to digestion. The final digest will contain:

Analyte	Concentration in Digest	Analyte	Concentration in Digest
Sb	50 ppb	Al	500 ppb
Ba	200 ppb	Be	10 ppb
Cr	50 ppb	Co	100 ppb
Cu	50 ppb	Fe	250 ppb
Mn	50 ppb	Ni	100 ppb
V	100 ppb	Zn	100 ppb
Ag	50 ppb		

Table based on final volume of 100 mL for water digests, and 200 mL for soil digests.

PDCA
SPIKES and LCSW

The LCSW for the PDCA extraction was make by adding 10 mL SPEX ICV-1™ solution to 100 mL of Type II water prior to extraction. The spike was made by adding 1000 µL of CLP SPK solution to a 100 mL aliquot of the sample prior to extraction.

ICP Analytes

Analyte	Concentration in Digest	Analyte	Concentration in Digest
Cd	50 ppb	Mn	500 ppb
Co	500 ppb	Ni	500 ppb
Cu	250 ppb	Ag	50 ppb
Fe	1000 ppb	V	500 ppb
Zn	500 ppb		

Graphite Furnace Analytes

Analyte	Concentration in Digest	Analyte	Concentration in Digest
Pb	500 ppb	Tl	2000 ppb
Se	2000 ppb		

Tables based on a final volume of 100 mL.

LAUCKS TESTING LABORATORIES, INC.
STANDARDS LOGBOOK

#	ANALYTE	ID or STOCK No.	STOCK CONC.	VOL/WT TAKEN	FINAL VOL.	CONC.	SOLVENT	PREP. DATE	INIT.	EXP. DATE	QC'D (Initials)
1	CLP.SPK	ME1.03.06 EXP 12/12/96	(CLPP SPK 1)	10mL	100mL		5% HNO_3 H_2O	03/28/96	RJH	12/12/96	
		ME1.03.07 EXP 12/12/96	(CLPP.SPK 2)								
		ME1.03.01 EXP 12/12/96	(CLPP.SPK 3)								
2	GF-CAL1 (Pb)	ME3.B6.09 EXP. 7/10/96	1000 ß	30µL	100mL	0.3 ppm	0.5% HNO_3 H_2O	03/28/96	RJH	05/30/96	
	(Se)	ME3.B6.10 EXP 7/10/96		50µL		0.5 ppm					
	(As)	ME3B1-9 EXP 5/30/96		100µL		1.0 ppm					
	(Tl)	ME3.B6.11 EXP 7/10/96		100µL		1.0 ppm					
3	GF-CAL2 (Pb)	ME3.B6.09 EXP 7/10/96	1000 ß	100µL	100mL	1.0 ppm	0.5% HNO_3 H_2O	03/28/96	RJH	05/30/96	
	(Se)	ME3.B6.10 EXP 7/10/96		100µL		1.0 ppm					
	(As)	ME3B1.9 EXP 5/30/96		250µL		2.5 ppm					
	3/28/96 RJH										

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-7202

Title: Metals Analysis Using Inductively Coupled Plasma - Mass Spectrometry (ICP/MS), SW 846 Method 6020

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UNCONTROLLED

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1. Introduction and Scope

1.1 Method Description

Inductively coupled plasma-mass spectrometry (ICP-MS) is a technique which is applicable to µg/L 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 acid preserved 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.

See Appendix V for reporting limits.
See Appendix I for analytical masses and standard concentrations.

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.

1.2 Sample Collection, Sample Storage, Holding Times

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 but need to be stored at 4° 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, which is not currently approved or done without specific client arrangement, the holding time is 28 days.

1.3 Definition of Terms

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 analysis group which uniquely identifies that batch. This number is preceded by an "A", mmddyy, ICPMS, matrix (W for water, S for soil), sequence number (i.e. A022595ICPMSW01).

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.

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".

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 are Sc45, In115, and Bi209.

Post-Digestion Spike - An analyte spike added to a portion of a prepared sampleshould 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 consitituent 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:

Perkin-Elmer ELAN 5000

2.2 Standards

SW 846 requires the use of one standard and a blank. Standards are made in a 1% HNO_3. See Appendix I for standard concentrations.

2.3 Internal Standards

Sc45, In115 and Bi209 are used as internal standards.

3. Safety precautions and Waste Disposal

3.1 Safety Precautions

All standards, samples and sample solutions should be handled as if they are hazardous substances.

Refer to the instrument manufacturer's manual for routine instrument precautions.

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.

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.

Radio Frequency - The ELAN 5000 uses a high energy RF. Although the generator is well shielded, care should be taken when operating the instrument. Pace makers can be adversely affected by exposure to high energy RF.

Plasma Radiation - Do not view the torch without the proper eye wear. Severe eye damage can occur if the plasma is viewed directly.

3.2 Waste Disposal

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

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.

4.2 Linear range study

Linear ranges for each analyte are determined by analyzing a high concentration "sample". The analytically determined concentration must be within 5% of the true value. The true value is the upper limit of the ICP/MS linear range. Linear ranges must be verified quarterly.

4.3 Internal Standards

A 50 uL aliquot of a 10 ppm stock solution of Sc45, In115 and Bi 209 is added to a 10 mL of all standards and samples prior to analysis.

Criteria

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.

Corrective action

If the criteria are not met, terminate the analysis, correct the problem, recalibrate, verify the new calibration, and re-analyze the affected samples.

Criteria

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.

Corrective action

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

Analyze standard solutions using a minimum of a calibration blank and one standard. The calibration curve must be verified by running an Initial Calibration Standard (ICV) and obtaining agreement within 10% of the expected concentration.

Criteria and Corrective Action:

Since a linear regression is not possible when using a two point calibration on the ELAN 5000, the standard curve is validated by evaluating the ICV and the subsequent CCVs. If the corresponding control limits for the ICV and CCV are exceeded, then the sample analysis must be discontinued, the cause determined and the instrument recalibrated. All samples following the last acceptable ICV, CCV must be reanalyzed..

4.5 Initial Calibration Verification

Immediately after the calibration curve, analyze a standard from a source other than that from which the calibration material was obtained.

Criteria

The calculated concentration of the ICV should be within 90%-110% of the true value.

Corrective action

If the ICV criteria are not met, the analysis is terminated. Perform system maintenance and re-calibrate the instrument.

4.6 Initial Calibration Blank

After the analysis of the ICV standard an instrument blank (ICB) is analyzed. The levels of target analytes in the ICB should not exceed the contract required detection limit .

Corrective action

If the initial ICB contains target analyte levels above the contract required detection 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 Blank (CCB)

A continuing calibration verification standard is analyzed after every 10 samples. Immediately following the CCV, a blank solution is analyzed. In addition, this standard and blank must be the last samples analyzed in the run.

Criteria

The CCV must fall within ± 10% of the true value.

The levels of target analytes in the CCB should not exceed the contract required detection limit.

Corrective action

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.

If the initial CCB contains target analyte levels above the contract required detection 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 CCBs which are within limits.

4.8 Interference Check Solutions A (ICSA) and AB (ICSAB)

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 are not recommended by the manufacturer for modern instruments. The ICSA and ICSAB solutions are prepared at different concentration levels from method 6020 to avoid clogging of the sampler cone orifice and damage to the instrument. Therefore, Al, Ca, Fe, Mg, Na, P, K, S, C, and Cl in the ICSA and the ICSAB are at 1/10 of the specified levels. See Appendix II for ICSA and ICSAB solutions concentrations.

(ICSA):

At the beginning and at the end of each run, an interference check solution A is analyzed. This solution contains interefering elements only. All other elements are not present in the solution. All elements not present should show a recovery of zero, or ± the contract required detection limit.

Corrective Action:
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.

(ICSAB):

At the beginning and end of each analytical sequence an ICSAB must be analyzed. Analytes must recover between 80-120%.

Corrective Action:

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

Method blanks are used to verify contamination free reagents and apparatus. They are prepared with every set of samples prepared at the same time or at least one blank every 20 samples, which ever is more frequent. Any analyte response above the CRDL is reported. For a method blank to be acceptable for use with the accompanying samples, the concentration of the blank of any analyte of concern should not be higher than the highest of either:

(1) 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.

Corrective Action:

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

The LCS is made from an independent source of the same matrix (soil or water) and is carried through the entire digestion procedure. An LCS is performed with each digestion batch. At a minimum, LCSW(water) 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.

Corrective Action

If the LCS is not within the required control limits, a redigestion will occur for the affected analytes.

4.11 Matrix Spike

A sample is chosen at random from the samples to be analyzed, and an aliquot of spiking solution is added to this sample prior to preparation. The analyst should attempt to avoid selecting samples which are identified by the client as blanks. As the purpose of the matrix spike is to test the system under "typical" conditions, the analyst may also avoid selecting the most difficult sample of the batch for spiking. The minimum frequency for MS analysis is 1 each per 20 samples per matrix. This will be best accomplished by running one with every batch for many analyses. This matrix spike sample is used to evaluate the matrix effect of the sample upon recovery of the analytes. The recovery of spike analytes is calculated as follows:

$$recovery,\% = \frac{(SS - S)}{SA} * 100$$

where:

SS = concentration in spiked sample
S = native concentration in unspiked sample
SA = spike added, the amount of spiking material actually added calculated on the sample basis.

For ICP/MS, control limits for spike recoveries will be 75-125% unless otherwise specified in the project specific QAPP. In-house control limits are based on historical performance. The recovery criteria are detailed in the QC Database QC_DB and will change from time to time.

Corrective Action:

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

A post digestion spike is also performed to a portion of a prepared sample. The minimum frequency for MS analysis is 1 each per 20 samples per matrix, control limits for post-spike recoveries will be 75-125%

Corrective Action:

Samples with post-spike recoveries outside control limits will be diluted and re-analyzed to compensate for matrix effects. The results must agree to within 10% of the origianal measured concentrations. A standard-addition technique may also be used to compensate for matrix effects.

4.13 Matrix Spike Duplicate/Sample Duplicate

Method QC consists of MS/MSD. A duplicate maybe be performed instead of a MSD. Other types of QC can performed at the client's request.

Criteria

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

For sample concentractions greater than 100 times the IDL, control limits for RPD of duplicates will be ±20% unless otherwise specified in the project specific QAPP. For sample concentrations less than 100 times the IDL, control limits for the difference will be ± the reporting limit. In-house control limits are based on historical performance. The RPD control limits are detailed in the current QC Database QC_DB and will change from time to time.

Corrective Action:

If a trend in out of control RPD values is observed, the methods used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable reproducibility. 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

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 100 times greater than the IDL, then results for those analytes will be flagged with an "E".

Corrective Action:

No corrective is necessary other than appropriately flagging the data.

5. Procedure

5.1 Instrumental Conditions

- Refer to manufacture's instruction for specific operating procedures. Allow at least 30 minutes for the instrument to stabilize before initiating any analysis.

- Conduct mass calibration and resolution checks in the mass regions of interest. The mass calibration and resolution parameters are required criteria which must be met prior to any sample being analyzed. The monitored masses of Mg, Rh, and Pb must meet the following criteria:

Element	RSD for replicate (minimum of four) integrations.	Mass, amu	Resolution @ 10 % peak height, amu
Mg	< 5%	23.90-24.10	<0.9
Rh	< 5%	102.80-103.00	<0.9
Pb	< 5%	207.90-208.10	<0.9

5.2 Analytical Operation

- Calibrate the instrument, using a calibration blank and a standard. Refer to Appendix I for the applied levels of concentration.

- All masses which could affect data quality are monitored to determine potential effects from matrix components on the analytes of interest.

- After the calibration has been established, an ICV solution 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.

- Analyze the interference check samples (ICSA and ICSAB) prior to and after the analysis of samples

- Analyze a CCV and a CCB once every 10 analytical samples.

- Dilute samples that exceed the established linear range of the instrument.

6. Reports

6.1 Data Packet Organization

- See the SOP metals validation for a check list detailing data packet organization
- If requested, all analysis performed under SW 846 guidelines the data can be reported via CLP SOW 3/90 forms.
- Data packages will be produced via Enviroforms. Analyte levels that are less than the MDL will be reported as the SDL followed by a "U". Analyte levels that fall between the MDL and the reporting limit will be flagged with a "B". Analyte levels greater than or equal to the reporting limit PQL will be reported without a flag.

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.

6.2 References:

Test Methods for Evaluating Solid Waste, SW-846, Method 6020, Revision 0, September 1994

Appendix I

Standard Solutions

Elements	Mass	STD, µg/L
Be	9	10
Na	23	500
Mg	26	500
Al	27	400
K	39	500
Ca	44	500
V	51	100
Cr	52	100
Mn	55	150
Fe	54,57	200
Co	59	100
Ni	60	400
Cu	63,65	50
Zn	66	200
As	75	100
Se	82	50
Ag	107	100
Cd	111	50
Sb	121	60
Ba	137	400
Tl	205	100
Pb	208	50

The STD is made by diluting 10.0 mL of the ICP/MS stock standard to a 100 mL final volume.

The ICP/MS stock standard which consists of:

100 µL	ICAL-1
1000 µL	ICAL-2
200 µL	ICAL-3
1000 µL	ICAL-4
100 µL	ICAL-5
200 µL	1000 ppm Sn

Note: 50 µL of 10 ppm Sc45, In115 and Bi209 is added to a 10 mL aliquot of standard.

Appendix II

ICSA and ICSAB Solutions

Element	ICSA (PPM)	ICSAB (PPM)
Aluminum	4	4
Arsenic		0.010
Cadmium		0.010
Calcium	12	12
Chromium		0.020
Cobalt		0.020
Copper		0.020
Iron	10	10
Magnesium	4	4
Manganese		0.020
Molybdenum		0.080
Nickel		0.020
Potassium	4	4
Selenium		0.010
Silver		0.020
Sodium	10	10
Vanadium		0.020
Zinc		0.010

Appendix III

QC Summary Table

Laucks Testing Laboratories

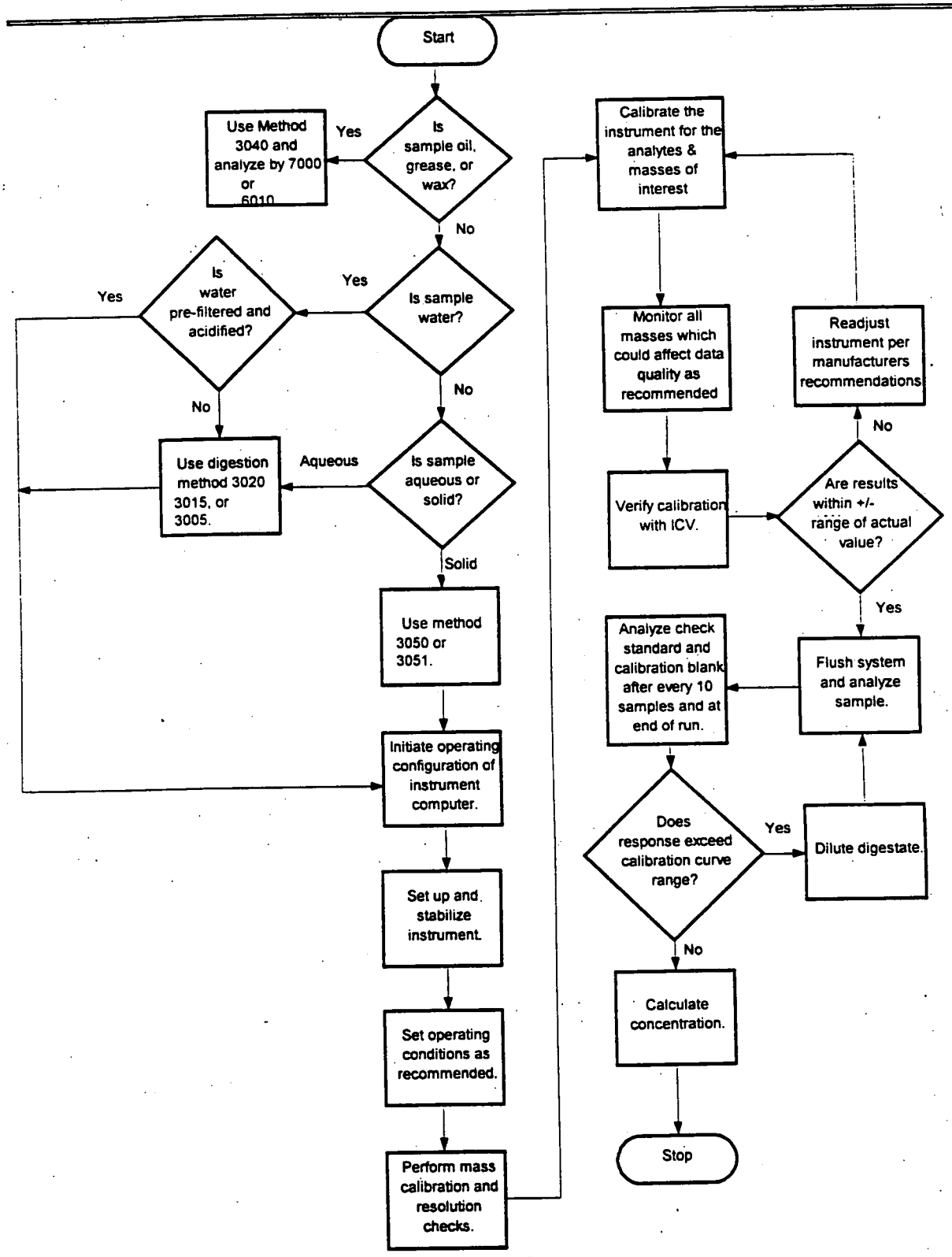
Method SW 846 6020 QA Requirements and Corrective Actions

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentatio
Mass Calibration Check	must be performed in mass regions of interest and be within ±0.1 amu of the actual value.	Mg 23.90-24.10 Rh 102.80-103.00 Pb 207.90-208.10	Beginning of each analysis.	Perform new mass calibration.	Instrument Logbook
Resolution Check	< 0.9 amu full width at 10% peak height	< 0.9 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 one standard	Beginning of each analysis	NA	In the raw data and/or on FORM 14.
Initial Calibration Verification	±10% of true value. Made from an independant source.	±10% of true value. Made from an independant 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 < 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 < 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	< 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	Documentatio
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, % Differenc	± 20% for analyte values greater than 100 times the IDL.	± 20% or current QC database criteria.	5% or per batch	reanalyze digestates, if still fail, consult QCO	Form 6, in raw data, or database repo
Matrix Spike Recovery		75-125% or current QC database criteria.	5% or per batch	Consult QCO	Form 5A, in raw data, or database repo
Post-Digestion Spike	75%-125%	75%-125%	5% or per batch	dilute and re-analyze or MSA	Form 5B, in raw data
Laboratory Control Sample	One/batch, no acceptance criteria	LCSW: 80%-120% LCSS: Manufacturer Specs.	One/batch	Redigest samples.	Form 7, in raw data, or database rep
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.

Appendix IV

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 3005.
No
Is sample aqueous or solid?
Aqueous
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 V

Routine Reporting Limits

SW 846 6020 Reporting Limits

Element	Reporting Limit, µg/L	Element	Reporting Limit, µg/L
Be	0.5	Cr	1.
Co	0.5	Ni	0.5
Cu	0.5	Zn	5.
As	1.	Se	1.
Ag	0.5	Cd	0.5
Mn	1.	Sb	1.
Ba	0.5	Tl	0.5
Pb	0.5	Zn	1.0

Reporting limits are approximately 2-10 times the instrumental MDL. The MDL is based on samples prepared using SW 846 3015. Values actually reported may be less than the routine reporting limits but above our method detection limit.

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-7501

Title: **Operation of the PE FIMS 400 for the Analysis of Hg**

Revision history:

Number	Date
1	02/04/98
0	02/14/96

Revised by: Bill Lundberg
Bill Lundberg, Inorganics Division Manager
Date: 2/5/98

Approved by: Harry Romberg
Harry Romberg, QC Officer
Date: 2-5-98

Approved by: Karen J. Kotz
Karen Kotz, Laboratory Director
Date: 2/5/98

UNCONTROLLED

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1. Introduction and Scope

1.1 Method Description:

1.1.1 This SOP covers the cold vapor analysis of Hg by the Perkin-Elmer Flow Injection Mercury System, Model 400 (FIMS 400). Sample handling and preparation are as required by SW 846 methods 7470 and 7471. This SOP is also valid for the preparation and analysis of samples under CLP protocol when used in conjunction with the SOW ILM04.0. Where the requirements of these procedures differ, the most stringent requirements are used. The requirement of analyzing soil samples in triplicate under 7471 is not used except on 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 adsorption tube through which passes light at a wavelength of 253.7 nm. The adsorption is then measured.

1.1.3 This method is restricted to use by, or under the supervision of analysts experienced in the technique described. Each analyst performing this method must have demonstrated the ability to perform the described analysis.

1.2 Sample Collection, Sample Storage, Holding Times

1.2.1 Samples are stored either in glass or plastic. The holding time is 28 days from collection. Soil samples are stored at 4° C±2° C. Water samples are stored at room temperature. Water samples are preserved by the addition of HNO_3 to a pH <2.

1.3 Definition of Terms

1.3.1 This section defines terms and acronyms as they are used in this SOP. Other terms, such as MS/MSD or method blank, are not defined here since it is assumed that the user of this SOP already understands their more general meaning.

1.3.2 LFB - Laboratory Fortified Blank - A sample of DIW to which a known amount of Hg is added.

1.3.3 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.

1.3.4 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.

1.3.5 CLP - Contract Laboratory Program - The USEPA program that contracts with laboratories to provide laboratory services. The term has come to mean a much broader set of methods and deliverables. In context of this SOP, CLP means procedures or operations which are detailed in the CLP contract and which are extended to a broader working definition.

1.3.6 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

1.3.7 DIW - Deionized water - Lab reagent water. This water should be free of virtually all analytes.

1.3.8 ICB - Initial calibration blank - This term is borrowed from CLP. An instrument blank is made up in the same way as calibration standards, without target analytes.

1.3.9 ICV - Initial calibration verification - is a standard which is analyzed at the start of each analytical run that is compared to the initial multi-point calibration to determine whether the instrument calibration is accurate. This verification standard is from a source different from that used to make the calibration standards

1.3.10 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.

1.3.11 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.

1.3.12 MDL standard - Method detection limit standard - A standard prepared so that the concentration of Hg is 1-5 times the anticipated MDL

1.3.13 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.

1.3.14 RSD or %RSD - Relative standard deviation or percent relative standard deviation - The ratio of the standard deviation of a set of values to the mean of the set of values. A measure of the similarity of the values one to another.

1.3.15 PBW - A blank carried through the sample preparation and digest procedures.

2. Equipment, Standards and Reagents

2.1 Equipment

2.1.1 Equivalent equipment may be used where appropriate.

FIMS 400 automatic Hg analyzer.
Perkin Elmer AS-90 autosampler.
Epson LQ300 dot-matrix printer.
Wells 18"X24" hot water bath.
BOD bottles
Analytical balance
Micropipettes: 25, 50, 100, 200, 500 and 1000 µl.
50 mL and 15 mL centrifuge tubes - polystyrene.

2.2 Standards

2.2.1 All standards must be prepared in 0.15% HNO_3.

2.2.2 Intermediate Hg solution: Dilute 100 µl of 1000 ppm Hg standard solution to 100 mL in DIW to which 0.15 mL HNO_3 has been added. The identification number of the standard solution used and the dilution must be entered on the digestion log (Appendix II). This solution must be prepared daily. Since the intermediate and working standards are prepared daily, they are not recorded in the standards logbook.

2.2.3 0.2 µg/L standard - Place 100 mL DIW in a BOD bottle. Add 20 µl stock Hg solution

2.2.4 0.5 µg/L standard - Place 100 mL DIW in a BOD bottle. Add 50 µl stock Hg solution.

2.2.5 1.0 µg/L standard - Place 100 mL DIW in a BOD bottle. Add 100µl stock Hg solution.

2.2.6 2.0 µg/L standard Place 100 mL DIW in a BOD bottle. Add 200 µl stock Hg solution.

2.2.7 5.0 µg/L standard - Place 100 mL DIW in a BOD bottle. Add 500 µl stock Hg solution.

2.2.8 10.0 µg/L standard - Place 100 mL DIW in a BOD bottle. Add 1000 µl stock Hg solution.

2.2.9 ICV solution - Add 100 mls DIW to a BOD bottle. Add sufficient stock ICV solution to produce a concentration of 1.0 to 5.0 µg/L.

2.2.10 CCV/CCB/LFB - In setting up the run, use the 5.0 µg/L standard for the CCV and LFB; for the CCB use the calibration blank.

2.2.11 MS/MSD - Add 100 µl intermediate Hg solution to the samples being spiked. When performing Hg under CLP protocols use 100 µl intermediate Hg solution and perform MS/Duplicate.

2.2.12 LCSS - Laboratory control soil sample. Concentrations and limits are certified by the manufacturer.

2.3 Reagents:

2.3.1 DIW - deionized water free of impurities.

2.3.2 H_2SO_4 (sulfuric acid) - concentrated, reagent grade

2.3.3 HNO_3 (nitric acid) - concentrated, reagent grade

2.3.4 HCl (hydrochloric acid) - concentrated, reagent grade

2.3.5 Stannous chloride - Add 11 g. $SnCl_2 \cdot 2H_2O$ (stannous chloride dihydrate) to 1 L DIW to which has been added 30 mL concentrated HCl.

2.3.6 $KMnO_4$ (potassium permanganate) - Dissolve 100 g. $KMnO_4$, marked "suitable for Hg determination", in 2 L DIW. This is a saturated solution. Sufficient undissolved $KMnO_4$ should be allowed remaining in the bottom of the bottle to assure a saturated solution.

2.3.7 $K_2S_2O_8$ (potassium persulfate) - Dissolve 50 g. $K_2S_2O_8$, marked "suitable for Hg determination", in 1 L DIW. This is a saturated solution. Sufficient undissolved $K_2S_2O_8$ should be allowed remaining in the bottom of the bottle to assure a saturated solution.

2.3.8 $NaCl/NH_2OH \cdot HCl$ (sodium chloride/hydroxylamine hydrochloride) - Dissolve 240 g NaCl (table salt is generally used) and 240 g. $NaCl/NH_2OH \cdot HCl$ (reagent grade marked "suitable for Hg determination") in 2 L DIW. Contamination has been experienced with this solution. For this reason a 50% solution should be analyzed by the FIMS 400 whenever a new bottle is made up. If the absorbance (peak height) exceeds .004 the container of $NaCl/NH_2OH \cdot HCl$ should be marked "Contaminated" and disposed of.

2.3.9 Carrier solution - The carrier is a 3% HCl solution.

3. Safety Precautions and Waste Disposal:

3.1 Safety Precautions

3.1.1 All standards, samples and sample solutions should be handled as if they are hazardous substances.

3.1.2 Refer to the instrument manufacturer's manual for routine instrument precautions.

3.1.3 Routine precautions include an awareness of the moving parts on the instrument you're using. These parts are often charged with power from an electrical component or with high pressure gas and have the potential to do harm if not used properly.

3.1.4 Electrical shock - All instruments present the possibility of electrical shock The operator should take all precautions including ensuring that all instruments are operated with fully grounded power outlets, turning off the instrument and disconnecting the instrument from the electrical power supply before working on any electrical components, etc.

3.1.5 Because of the toxic nature of Hg vapor, inhalation or skin contact should be avoided.

3.1.6 The usual precautions should be taken in handling acids.

3.1.7 $SnCl_2$ is a skin and eye irritant; avoid contact.

3.1.8 The addition of hydroxylamine after digestion releases gas, including chlorine. Avoid inhalation; use hood if necessary.

4. Calibration and Quality Control

4.1 Method Detection Limit Study (MDL).

4.1.1 MDL studies for Hg water samples and soil samples are performed annually. Consult the current SOP on MDL determinations for specifics.

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 made fresh daily from the intermediate standard in 0.15% HNO_3.

4.2.2 The calibration blank, the ICV, the LCS, the LFB, the ICB and all standards are digested along with the samples being analyzed.

4.3 Calibration Criteria

4.3.1 Calibration curve - CC must be greater than 0.995.

4.3.2 ICV - For CLP analyses the limits are 80-120 %, for SW 846 the limits are 90-110%.

4.3.3 ICB - Limitations presently in use are -0.2 µg/L to 0.2 µg/L .

4.3.4 A PBW and an LFB are run immediately following the successful calibration. The limitations on the PBW are the same as the limitation on the ICB. The limitations on the LFB are as set forth in QC-DB.

4.3.5 If the above criteria are not met, recalibration must be performed.

4.4 Continuing quality control:

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 count as samples for this purpose.

4.4.2 Criteria - For CCV's the limitations are 80% to 120%. For blanks the limitations are -0.2 µg/L to 0.2 µg/L .

4.4.3 Corrective action - If a CCV or CCB are out of control, a recalibration must be performed followed by a reanalysis of all samples since the last previous successful CCV and/or CCB.

4.5 Matrix Spike

4.5.1 A sample is chosen at random from the samples to be analyzed, and 100 µl of intermediate standard is added. The spike level is then 1.0 µg/L. The analyst should attempt to avoid selecting samples which are identified by the client as blanks. As the purpose of the matrix spike is to test the system under "typical" conditions, the analyst may also avoid selecting the most difficult sample of the batch for spiking. It is not always required that a matrix spike analysis be performed with each preparation/analysis batch, however, the minimum frequency for MS analysis is 1 each per 20 samples per matrix. This will be best accomplished by running one with every batch for many analyses. This matrix spike sample is used to evaluate the matrix effect of the sample upon recovery of the analytes. The recovery of spike analytes is calculated as follows:

$$\text{recovery,\%} = \frac{(SS - S)}{SA} * 100$$

where:
SS = concentration in spiked sample
S = native concentration in unspiked sample
SA = spiked added, the amount of spiking material actually added to the sample calculated on the sample basis

4.5.2 The recovery criteria are listed in QC_DB and will change from time to time.

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 SRM and/or blank spike analysis. In all cases a narrative explanation of the condition is required to detail the corrective actions taken.

4.6 Matrix Spike Duplicate

4.6.1 The compound recovery criteria are identical to those for the matrix spike sample. In addition, the matrix spike duplicate is used measure method precision. This is done by computing the relative percent difference (RPD) between the matrix spike and matrix spike duplicate recovery values. This calculation is as follows:

$$RPD = \frac{|S1 - S2|}{(S1 + S2)/2} * 100$$

where:
S1 = measured concentration for MS sample
S2 = measured concentration for MSD sample

4.6.2 RPD control limits are listed in QC_DB and will change from time to time.

4.7 Sample Duplicate

4.7.1 Sample duplicates are required when CLP practices are employed, or when the method specifically calls for duplicates. At least one duplicate sample per 20 samples per matrix is required when matrix spikes are being performed.

$$RPD = \frac{|S1 - S2|}{(S1 + S2)/2} * 100$$

where:
S1 = measured concentration in the initial analysis
S2 = measured concentration in the duplicate analyses

4.7.2 The RPD control limits listed in QC_DB and will change from time to time.

4.7.3 Corrective action

4.7.3.1 If a trend in out of control RPD values is observed, the methods used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable reproducibility. Generally, if recoveries are in control and no analyte of interest was detected in any of the samples, no immediate action will be taken on that

sample set. If integrity of reported sample values is in doubt, re-analysis may be called for. Corrective actions should be discussed with the Quality Control Officer.

5. Operation Procedures:

5.1 Digest - BOD bottles - Soil or Water:

5.1.1 Digestion log (Appendix II): The digestion log should show the sample numbers, the date, the name of the analyst, the time placed in the hot water bath, the time when the temperature reached 95± 5° C, and the time removed from the hot water bath. One should be prepared for each run. An example is attached as Appendix II. The numbers of each BOD bottle must be placed in the leftmost column of this form to identify which sample is in each BOD bottle.

5.1.2 Preparation of standards:

5.1.2.1 Prepare an intermediate standard daily by adding 100 µl of the 1000 mg/L stock standard to 100 mL of 0.15% HNO_3 in DIW, giving a concentration of 1.0 mg/L of Hg.

5.1.2.2 Place 100 mL DIW in each of 9 BOD bottles.. Mark these BOD bottles with the concentration of each standard in µg/L , plus one marked "Blank", one marked "ICV" and one more marked ICB.

5.1.2.3 Add from the intermediate standard the following amounts to the following BOD bottles:

0 µg/L	0 µl
0.2 µg/L	20µl
0.5 µg/L	50 µl
1.0 µg/L	100 µl
2.0 µg/L	200 µl
5.0 µg/L	500 µl
10.0 µg/L	1000 µl

5.1.2.4 To the ICV BOD bottle add that amount of the ICV standard which will produce a concentration from 1.0 µg/L to 5.0 µg/L .

5.1.2.5 The standards, the ICV and the ICB must be digested along with the PBW, LFB and samples.

5.2 Digestion - Water Samples

5.2.1 Place 100 mL DIW in the PBW BOD bottle.

5.2.2 Place 100 mL DIW + 100 µl intermediate standard in the LFB BOD bottle.

5.2.3 Place 100 mls sample in the BOD bottle designated for that sample, being sure to place 100 mL sample in the designated duplicate, MS and MSD BOD bottles for the sample involved. TCLP samples and spikes should be diluted 20 mL/100 mL (dilution factor of 5) in DIW. The TCLP spikes should be diluted 2 mL/100 mL in DIW.

5.2.4 Add the required quantity (See Sec. 4.5.1) of intermediate standard to the MS and MSD BOD bottles.

5.2.5 Add the following reagents to each BOD bottle:

5 mL concentrated H_2SO_4
2.5 mL concentrated HNO_3
15 mL $KMnO_4$ solution
8 mL $K_2S_2O_8$ solution

5.2.6 The $KMnO_4$ color must persist for 15 minutes. If it does not, more $KMnO_4$ must be added.

5.2.7 Place all the BOD bottles, being careful to keep them in order, in the hot water bath in a hood, and record the time on the digest log. Monitor the temperature of the hot water bath and record the time when the temperature reaches 90° C. Continue to heat the water bath, keeping the bath at 95 ± 5 C for two hours.

5.2.8 Remove the BOD bottles from the hot water bath and allow to cool.

5.2.9 Add 6 mL $NaCl/NH_2OH \cdot HCl$ solution and mix until the mixture clears. If some undissolved permanganate remains, it will usually disappear on standing after a few minutes.

5.3 Digestion - Soil samples:

5.3.1 Place 100 mL DIW in the PBW BOD bottle.

5.3.2 Place approximately 0.5 g., or, for certain CLP and 846 analyses, approximately 0.2 g of sample in each of the BOD bottles to which that sample applies. Record the exact weight on the digestion log.

5.3.3 Add 3.75 mL HCl , 1.25 mL HNO_3 and 5 ml DIW to each BOD bottle.

5.3.4 Place all BOD bottles in a hot water bath in a hood that is at 95° C ± 5° C. Maintain the heat at this level for 2 minutes.

5.3.5 Cool for a few minutes and add 50 mL DIW and 15 mL $KMnO_4$. Mix.

5.3.6 The $KMnO_4$ color must persist for 15 minutes. If it does not, more $KMnO_4$ must be added.

5.3.7 Place all BOD bottles in a hot water bath that is at 95° C ± 5° C. Record the time on the digest log. Maintain the hot water bath at this temperature for 30 minutes. Remove the BOD bottles and allow to cool.

5.3.8 Add 6 mL $HCl/NH_4NH_2OH \cdot HCl$ solution and mix to remove color.

5.3.9 Add 50 mL DIW. Allow to cool.

5.4 Analysis on FIMS 400.

5.4.1 This is a programmed analysis. The method name is "hg_cv" (Appendix V). This is a 6 point curve with a blank correction. The development of the calibration curve, including the ICV and ICB, is automatic. If the calibration is unsuccessful, recalibration is attempted. If a standard is seriously out of line, it is ignored by the Perkin-Elmer software. 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 recalibrates and reruns all samples since the last preceding valid CCV or CCB. A CCV and a CCB are also run after the last sample in the run.

5.4.2 Sample Information sheet - Appendix IV.

5.4.2.1 Click the automatic analysis button on the screen which appears after activating the program.

5.4.2.2 Click the "Sample Information" button on the toolbar.

5.4.2.3 A blank sample information sheet appears. Previous sample information sheets may be obtained by activating the File - Open menu, designating "Sample Information" from the dialog which appears. The sample information sheet should be filled in to tell the computer which auto-sampler locations to go to.

5.4.2.4 Sample ID column - Some typing can be saved by double clicking on the column title. On the resulting form the job order may be typed in and the sample numbers will then be incremented by 1 for the autosampler locations indicated. The sample ID's will then be filled in. Sample: "9802094-0001..0002. .".

5.4.2.5 Sample weight column - For soil samples the weight of the samples should be filled in. At the top, the volume units should be filled in "mL", the weight units "g".

5.4.2.6 Sample units column - Typing may be saved by double clicking on the column title and indicating the units on the dialog box which appears, and indicating the AS locations involved. In the case of water samples this will ordinarily by "µg/L ". For soil samples the button "weight:weight" should be clicked and the desired units specified in the dialog box selected. If the dialog box has been properly filled in, the machine is programmed to compute the proper units (e.g. mg/kg). It should be kept in mind that this is on an as received basis, not a dry basis.

5.4.2.7 User dilution column - Where a dilution is involved (e.g. TCLP analyses) the dilution factor should be entered. As in the sample weight and sample units columns, above, typing may be saved by double clicking the column title.

5.4.2.8 The autosampler tray being used at present is "Tray B".

Sampler Location	Size	Contents
1	50 mL tube	Calibration Blank
2	50 mL tube	0.5 µg/L standard
3	50 mL tube	1.0 µg/L standard
4	50 mL tube	2.0 µg/L standard
5	50 mL tube	5.0 µg/L standard
6	50 mL tube	10.0 µg/L standard
7	50 mL tube	ICV
8	50 mL tube	ICB
9	15 mL tube	PBW or LCSS
10	15 mL tube	LFB or sample
11-109	15 mL tubes	Samples

5.4.3 All tubes should be marked with their contents before being filled. Then the tubes are filled from the BOD bottles and placed in the autosampler locations designated either by the method or by the sample information sheet.

5.4.4 At the beginning of each day of analysis, the following steps are taken to prepare the instrument:

5.4.4.1 The flows of the carrier solution and the reductant solution (the stannous chloride solution) are measured by the methodology suggested in the Perkin-Elmer handbook "Setting Up and Performing Analyses", 2.4.2-2.4.4. The rinse container (autosampler location 0) is rinsed and filled with 3% HCl solution. This is then analyzed using the location button in the analysis window of the automated analysis window, and the curve produced displayed through the curve button in the toolbar. The curve should be flat and less than 0.0001 absorbance units.

5.4.5 Analysis - The analysis is ordinarily started by clicking the "Analyze All" button in the Automated Analysis window. If only calibration is desired, the "Calibrate" button may be used. If samples are to be analyzed by an already existing calibration curve use the "Analyze Samples" button.

5.4.5.1 Once the analysis has been started the autosampler places the sampling probe in the sample/calibration cup. The peristaltic pump begins pulling sample through the sample loop. After the line has been purged, the injection valve cycles, and injects the sample into the carrier flow. This flow is then combined

with the SnCl2 and mixed. Next it goes to a liquid/vapor separator. The liquid goes to waste and the vapor is transported into the adsorption cell of the spectrophotometer. All samples and standards are measured in duplicate.

5.4.6 Run log - A run log will be printed as the analysis progresses. The print program is designed to transmit the data to be printed to the printer one page at a time. Thus several autosampler locations will be analyzed before anything appears on the printer. If anything else is printed (e.g. the sample information sheet) while the automated analysis window is open, the computer must be rebooted, or a run log will not be printed.

5.4.7 Additional samples may be analyzed after the run designated on the sample information sheet by using the "Select Location" button on the "Analyze" page of the Automated Analysis window. The autosampler locations should be filled in the resulting dialog.. The instrument will then analyze the samples indicated. This may be used to reanalyze the samples with questionable results, or to analyze diluted samples.

5.4.8 Dilution - Where analysis results are above the high standard (10.0 µg/L) an aliquot of the sample diluted with the calibration blank may be analyzed as set forth in 5.4.7 above.

6. Reports

6.1 Data Packet Organization

6.1.1 A report is prepared using the reformat procedure on the main menu. The reformatted design is entitled "Summary". It is transferred to a floppy disk, which is then imported to a spreadsheet using Microsoft Excel 5.0. This must then be formatted into the form included in Appendix III.

6.1.2 The final data packet is to include the following:

1) The report (See Appendix III).
2) The QC_DB Report Form
2) The digestion log (Appendix II).
4) The run log, if required.

6.1.3 All results for quality control tests are entered into QC_DB. A summary report of all data entered must be included in the data packet. The routine minimum is a method blank report, an MS/MSD or MS/duplicate report and a SRM/LSCW.

6.2 Data Qualifying Flags

Flag	Definition
U	The analyte of interest was not detected, to the limit of detection indicated.

6.3 Control charts

6.3.1 The recovery values for the LCS/SRM are plotted on control charts.

Appendix I - QC Summary Table

Laucks Testing Laboratories
Method 245.1,7471,7470 QA Requirements and Corrective Actions

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentatio
Initial Calibration	CC > 0.995	CC > 0.995	once per batch	recalibrate	run log
Initial Calibration Verification	90-110 % SW846 80-120% 245.1	Per the method	one per 20	recalibrate	run log, report
Initial Calibration Blank	Below R.L.	Below R.L.	one per sequence	recalibrate	run log, report
Continuing Calibration Verification	80-120 %	80-120 %	At the beginning,every 10 samples and after the last sample	recalibrate and rerun samples since last CCV	run log
Continuing Calibration Blank	Below R.L.	Below R.L.	At the beginning,every 10 samples and after the last sample	recalibrate and rerun samples since last CCB	run log
Matrix Spike Recovery	SW-846 See QC_DB. CLP 75-125%	See QC_DB	Every 20 samples	Consult QC officer. Flag data or reanalyze.	QC_DB
MS/MSD RPD	See QC_DB	See QC_DB	Every 20 samples	Consult QC officer. Flag data or reanalyze.	QC_DB
Duplicate % Difference	20% RPD CLP	See QC_DB	Every 20 samples	Consult QC officer. Flag data or reanalyze.	QC_DB

Laboratory Fortified Blank (LFB)	85-115 %	See QC_DB	Once per 20	Recalibrate	Run log
Standard Reference Material (SRM) Recovery	Soil Manufacture's limits Water See ICV	Manufacturer's limits	Once per 20	Recalibrate	Run log

Appendix - II - Sample Digestion Log

Date		Bottles into water bath (time)	
Analyst		Water bath at 95°C (time)	
Standard ID	ME-	Bottles out of water bath (time)	
ICV No	ME-	Spike = 100 µl of Std	

No.	Sample ID	Sample size g , mL	Final volume,mL	Comments	Bottle #
	S0 ppb		100 ml		
	S0.2 ppb 20 µl of Std		100 ml		
	S0.5 ppb 50 µl of Std		100 ml		
	S1.0 ppb 100 µl of Std		100 ml		
	S2.0 ppb 200 µl of Std		100 ml		
	S5.0 ppb 500 µl of Std		100 ml		
	S10.0 ppb 1000 µl of Std		100 ml		
	ICV		100 ml		
	ICB		100 ml		
	LCSW		100 ml		
	CRA		100 ml		
	PBW		100 ml		
			100 ml		
			100 ml		
			100 ml		
			100 ml		
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			100 ml		
			100 ml		
			100 ml		

$NH_2OH \cdot HCl$	NaCl	$K_2S_2O_8$	$SnCl_2 \cdot 2H_2O$	$KMnO_4$
REA1-6-3	REA1-7-9	REA1-5-7	REA1-5-3	REA1-5-4

Appendix III - Example Report Form

Hg in Water and TCLP
Digested 1/7/98
Analyzed 1/8/98

Sample	Time	Value		% Rcvry	RPD	Flag	Comment
Calib Blank	11:13:29		µg/L				
S0.2	11:16:23		µg/L				
S0.5	11:19:19		µg/L				
S1.0	11:22:19		µg/L				
S2.5	11:25:17		µg/L				
S5.0	11:28:15		µg/L				
S10.0	11:31:16		µg/L				
ICV1 ME7-23-2=4	11:34:21	4.028	µg/L	101%			
ICB	11:37:21	0.001	µg/L				
CCV1=5	11:40:16	5.015	µg/L	100%			
CCB1	11:43:10	0.007	µg/L				
LCSW1	11:46:04	4.067	µg/L	102%			
CRA	11:48:57	0.226	µg/L				
PBW	11:51:55	0.003	µg/L				
12611-11	11:54:50	2.215	µg/L				did not report
12679-02	11:57:47	0.007	µg/L				
12679-02dup	12:00:44	0.017	µg/L				
12679-02ms Spk=1	12:03:42	12.842	µg/L	1284%		N	see below
12679-03	12:06:42	0.083	µg/L				
12679-04	12:09:34	-0.016	µg/L				
CCV2=5	12:12:28	5.138	µg/L	103%			
CCB2	12:15:24	0.013	µg/L				
12679-05	12:18:16	0.026	µg/L				
12679-06	12:21:11	0.029	µg/L				
12679-07	12:24:04	0.002	µg/L				
12679-08	12:26:58	8.239	µg/L				see below
12679-09	12:29:52	23.125	µg/L				did not report
12679-10	12:32:46	0.209	µg/L				did not report
12725-01	12:35:44	0.367	µg/L				
12725-02	12:38:39	-0.226	µg/L				
12725-02dup	12:41:36	-0.419	µg/L				
CCV3=5	12:44:32	4.945	µg/L	99%			
CCB3	12:47:26	0.014	µg/L				
12725-02ms Spk=200	12:50:25	199.903	µg/L	100%			
12184-5	12:53:22	1.436	µg/L				
12184-5dup	12:56:18	1.433	µg/L				
CCV4=5	12:59:16	5.111	µg/L	102%			
CCB4	13:02:10	-0.028	µg/L				
12679-02ms (rerun)	13:08:05	0.959	µg/L	96%			
12679-02ms (10x dil)	13:10:59	-0.646	µg/L				did not report
12679-08 (rerun)	13:13:49	0.001	µg/L				
12679-09 (10x dil)	13:16:40	0.211	µg/L				did not report
CCV5=5	13:19:35	4.940	µg/L	99%			
CCB5	13:22:32	0.011	µg/L				

Appendix IV - Example Sample Information Sheet

Autosampler Loading List

Sample Information File: 0205985.SIF
Methods: Soils

Location	Elements	Solution
0	Hg	Wash Solution
1	Hg	Calib Blank
	Hg	CCB1: 0.0000 µg/L
	Hg	CCB: 0.0000 µg/L
2	Hg	S0.2: 0.2 µg/L
3	Hg	S0.5: 0.5 µg/L
4	Hg	S1.0: 1.0 µg/L
5	Hg	S2.0: 2.0 µg/L
6	Hg	S5.0: 5.0 µg/L
	Hg	CCV1: 5.0000 µg/L
	Hg	CCV: 5.0000 µg/L
7	Hg	S10.0: 10.0 µg/L
8	Hg	ICV: 4.0000 µg/L
9	Hg	ICB: 0.0000 µg/L
10	Hg	Sample: TRA
11	Hg	Sample: PBW
12	Hg	Sample: LCSS
13	Hg	Sample: 9801614-01
14	Hg	Sample: 614-01ms
15	Hg	Sample: 614-01msd
16	Hg	Sample: 549-02 (10x)
17	Hg	Sample: 549-03 (10x)
18	Hg	Sample: 549-03(20x)

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

Written by: _____ Date: 2/2/98

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UNCONTROLLED

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1. Introduction and Scope

1.1. Method Description

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.

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.

This procedure describes the methodology used to establish retention time windows for chromatographic methods. It is based on the practice outlined in SW846, Method 8000. In general terms, 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.

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. RTW (Retention Time Window)

The width, in minutes, of the retention time window half width. The retention time window for identification is ±RTW.

3. Equipment list and standards

3.1. Equipment

3.1.1. Chromatographic system

The same system that will be used for the analysis of samples or sample extracts. 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

Calibration standards required by the method.

4. Safety precautions and waste disposal

4.1. Safety precautions

4.1.1. Standards, samples, and samplesolutions

Handle as if they are hazardous substances.

4.1.2. Instrument operation

Refer to the instrument manufacturer's manual for routine instrument precautions.

Routine precautions include an awareness of the moving parts on the instrument you're using. These parts are often charged with power from an electrical component or with high pressure gas and have the potential to do harm if not used properly.

4.1.3. Electrical shock

All instruments present the possibility of electrical shock. The operator should take all precautions including ensuring that all instruments are operated with fully grounded power outlets, turning off the instrument and disconnecting the instrument from the electrical power supply before working on any electrical components, etc.

4.2. Waste disposal

4.2.1. All waste disposal precautions and procedures will be 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

Calibrate the instrument as described in the analysis SOP. Before establishing windows, make sure that the chromatographic system is operating under optimal conditions.

6. Operation procedures

6.1. Data collection

For the data collected over the course of at least one analytical sequence no less than 72 hours long, tabulate the analyte retention times for all method target analytes and surrogate compounds from all standards analyzed.

> **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 which more closely models real world operating conditions.

To mimic real world operating conditions the standards must bracket sample extracts just as in a normal sample analysis sequence. Collect data for a minimum of 3 standards in each sequence. For multi-response analytes such as Aroclors, select the same peaks which will be used for compound identification.

Record the retention time for each single component target analyte, surrogate spiking compound, and multi-component representative peak to 3 decimal places.

6.2. Calculations

Compute the mean and standard deviation of the measured retention times for each compound using the following equations.

6.2.1. Mean

$$\bar{t} = \frac{\sum_{1}^{n} t_i}{n}$$

6.2.2. 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

6.2.3. Retention time window

$$\text{RTW} = \bar{t} \pm 3 * SD$$

6.2.4. Frequency of RTW determination

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.

6.2.5. Constraints on the experimental determination of RTWs

6.2.5.1. RTW too small

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, to the limits of the chromatography system. In that 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.

6.2.5.1.1. 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.

6.2.5.1.2.Method 2

Set the RTW using the following guidelines.

Run Type	RTW half-width, minutes
Narrow bore, megabore capillary	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 methodology, the RTWs are fixed in the Statement of Work. This methodology will not apply to pesticides/PCBs determined using the CLP SOW.

The default retention time window half-width (0.03 minutes) that is called out in SW-846 is 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 8000.

6.2.5.2. RTW too large

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

The analytical method for each SOP covers this in more detail, but the general methodology is as follows. After the analysis of either the initial multi-point calibration standards or 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.

7.2. Analyst discretion in analyte identification

Irrespective of the RTW established for analyte identification, the judgment of the analyst weighs heavily in the interpretation of chromatograms. Sample-specific effects can alter the observed retention times of target analytes in sample extracts. In such

cases, it is acceptable for the analyst to determine that the target analytes are outside the established RTWs.

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 must be completely documented in narrative comments for the sample set.

7.3. Corrective actions

The surrogate compounds added to each sample, blank, QC sample, and calibration standards 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 must be documented on a sample by sample basis and the corrective action used for compound identification documented.

The retention times 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

The appropriate analytical department will retain the original files of all RTW studies.

8.2. Working copies

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.

In addition, these retention time window study summaries will be signed off by the department manager and the summary report scanned and stored in LaserFiche.

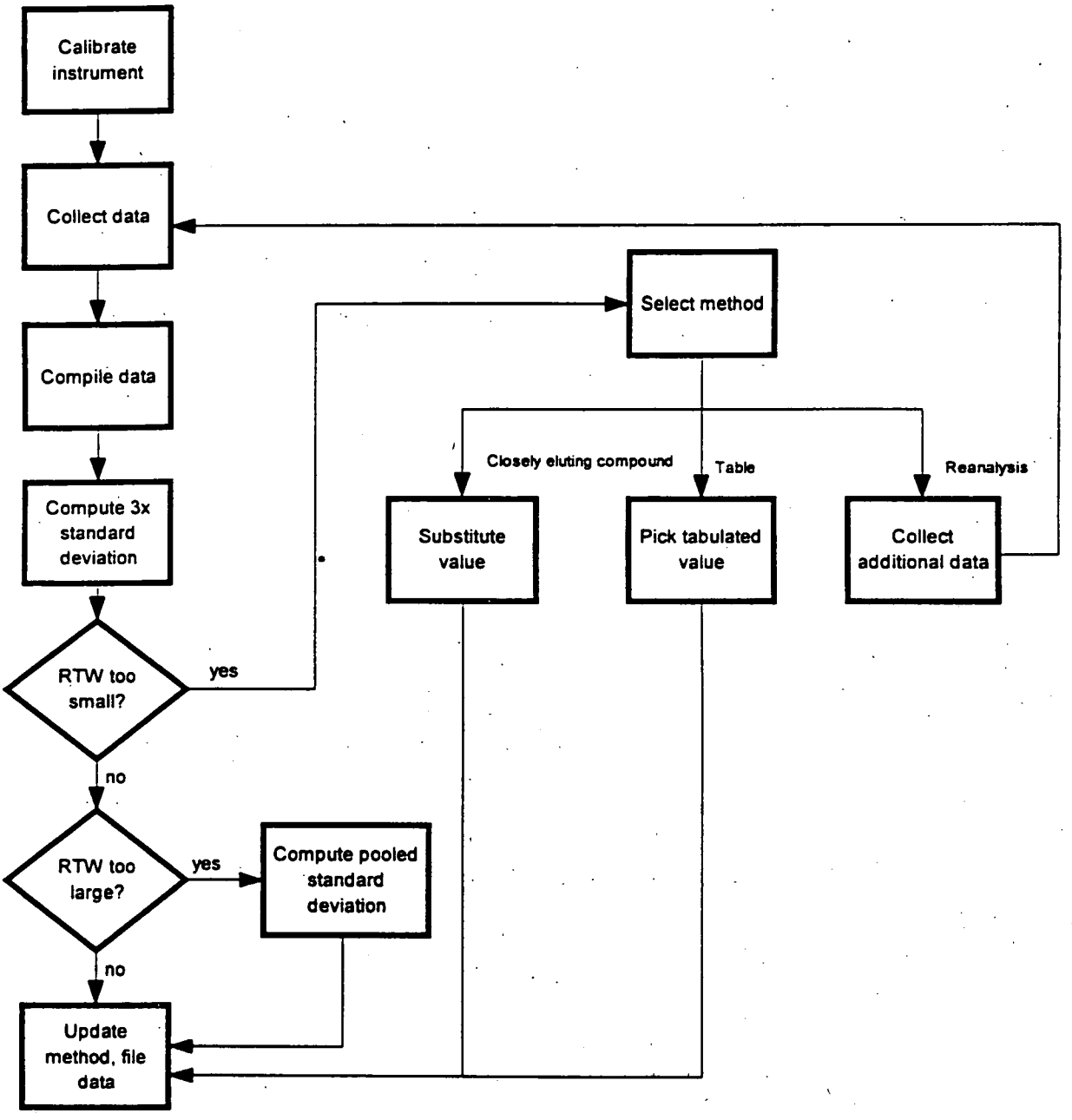
References:

John Keenan Taylor, *Quality Assurance of Chemical Measurements*, Lewis Publishers, 1989

USEPA, *Test Methods for Evaluating Solid Waste*, SW-846, 8000B, various analytical methods.

9. Appendix 1. Flow Chart

Method LTL-8000



LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-8265

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1. Introduction and Scope

1.1 Method Description

Method 8260 is used in the determination of volatile organic compounds in soil, sediment, aqueous, and other matrices. This SOP addresses the determination of volatile organics in different matrices, which include medium level soils as well as low level water samples. A 25 mL sample volume is employed in order to achieve lower detection limits for the low level waters. In addition, the instrument acquisition parameters have been added for the analysis of vinyl chloride by Selected Ion Monitoring (SIM) in Appendix III, and Appendix IV contains a tabular list of the non-routine Appendix IX analytes and their respective calibration ranges.

1.1.1 Because this method employs sample introduction via a purge and trap sample concentrator, it is applicable to volatile compounds that have boiling points below 200°C and that are insoluble or only slightly soluble in water. Compounds that are more soluble in water have elevated detection limits due to their decreased purging efficiency. These include, but are not limited to ketones, nitriles, acetates, acrylates and ethers.

1.1.2 This method is restricted to use by, or under the supervision of analysts experienced in the use of purge and trap systems, gas chromatography/mass spectroscopy and in the interpretation of chromatograms and mass spectral data. Each analyst performing this method must have demonstrated the ability to perform the described chromatographic analysis and data interpretation.

1.2 Method Deviations

1.2.1 Detailed below are any deviations from the published version of method SW 8260B. All deviations are followed as the standard operation procedure by Laucks Testing Labs.

1) In order to be more consistent and adhere to practical precision, Laucks uses method reporting limits for method blank criteria instead of method detection limits as referenced in SW 846. Common laboratory solvents, such as methylene chloride and acetone, are allowed to be present in the method blanks ≤ 5 times the RL. 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. This window is considerably narrower than the 0.06 RRT units specified in method SW 8260B and is considered more likely to ensure acceptable method performance.

3) The BFB tuning criteria used is taken from CLP SOW OLM03.1, which is not a deviation from the method since SW 8260B allows the use of alternative tuning criteria, e.g., CLP and/or 524.2 The tuning criteria are tabulated in section 4.4.5.

1.3 Sample Collection, Sample Storage, Holding Times

1.3.1 Waters

1.3.1.1 Samples are collected with zero headspace in 40 mL glass containers containing preservative (either HCl, H_2SO_4 or solid $NaHSO_4$) and sealed with Teflon-lined caps.

1.3.1.2 The water samples are preserved to a pH of ≤2, and all samples are stored at 4°C.

1.3.1.3 The holding time to analysis is 14 days from date of collection. The pH values for the water samples are measured subsequent to sample analysis. The pH values, when not preserved to a pH ≤2 are noted in the instrument logbook and the client report.

1.3.2 Soils

1.3.2.1 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. Therefore, when collecting 5 grams of sample, the VOA vial should contain 1 gram of sodium bisulfate. The sodium bisulfate is added in order to ensure a sample pH of ≤2. The vials used for low level soils should have the tared weight recorded on the label and sealed with septum-lined screw caps.

1.3.2.2 Another option is the use of the EnCore™ sampler, the Purge-and-Trap Sampler™, and a cut plastic syringe to transfer the sample to the soil vial. If the EnCore™ sampler is used in the field, the samples may be stored for up to 48 hours. However, samples collected in this device should be transferred to the soil sample vials (and preserved) as soon as possible, or analyzed within 48 hours. The holding time for soils is 14 days from the date of collection.

1.3.2.3 At the time of this writing, the 3rd update of the 3rd edition to SW 846 requires that high level soils be collected in using one of the following options:

1.3.2.3.1 Option 1: Samples will be collected in duplicate using 5 gram and/or 1 gram aliquots which are to be weighed out in the field. The soil samples are to be collected in a 40 mL vial containing methanol. The vials used for high level soils should have the tared weight recorded on the label and sealed with septum-lined screw caps.

1.3.2.3.2 Option 2: Samples will be collected using an EnCore type sampler or equivalent, and preserved at the laboratory.

1.3.2.3.3 Option 3: Samples will be collected in vials with zero headspace.

1.3.2.4 The practices in sections 1.3.2.1 and 1.3.2.2 will be implemented as SAPs, QAPs, agencies and clients require without updating this SOP until the practice is routine and this document is up for its annual review. Until this practice is routine and accepted by all agencies Laucks will continue to accept unpreserved soil samples for low or high level analysis. The samples must be collected in 2- or 4-oz glass containers and sealed with teflon-lined caps. These samples will be introduced by method 5030A and analyzed according to this SOP. Please note that for South Carolina samples, Method 5035 must be followed.

1.3.2.5 The holding time to analysis is 14 days from date of collection.

1.4 Definition of Terms

1.4.1 This section defines terms and acronyms as they are used in this SOP. Other terms, such as MS/MSD or method blank, are not defined here since it is assumed that the user of this SOP already understands their more general meaning.

Blank Spike	A background free matrix (DIW for water, clean sand for soils/sediments) to which known amounts or target analytes and surrogates are added each time samples are analyzed. Blank spikes are required on all HAZWRAP and NFESC work. In the context of this SOP, a blank spike is the same as a QC check standard. See also QC check standard.
CCC	Calibration check compound. There are 6 analytes which must meet the minimum %RSD of 30% in the initial calibration and a %D of 20% in the continuing calibration standard to validate linearity. These analytes are 1,1-dichloroethane, chloroform, 1,2-dichloropropane, toluene, ethylbenzene and vinyl chloride.

CCV	Continuing calibration verification. This is a standard analyzed at some prescribed frequency during the analysis sequence to determine whether the instrument has remained in calibration.
CLP	Contract Laboratory Program. The USEPA program that contracts with laboratories to provide laboratory services. The term has come to mean a much broader set of methods and deliverables. In the context of this SOP, CLP means procedures or operations which are detailed in the CLP contract and which are extended to a broader working definition.
IBLK	Instrument blank. An instrument blank is PFW containing the method surrogates and is introduced into the instrument to monitor for carry over between sample analyses.
IDL	Instrument detection limit. The lowest concentration of a target analyte that will yield a signal:noise ratio of at least 3x. Used as a starting point for selecting MDL study spiking levels.
MDL	Method detection limit. The lowest concentration in a sample which will yield a positive result that is greater than zero at a known level of confidence. MDLs are empirically determined by Laucks.
PFW	Purge-free water. Laboratory deionized water that is boiled for 30 minutes prior to use. The systems used to provide deionized water at Laucks all contain carbon polishing filters which are capable of providing purge-free water for use as reagent water.
QC Check Standard	Quality control check standard. Referred to in this SOP as a blank spike. A QC check standard is a requirement of SW 846 method 8000 and is used to determine whether the analytical system is in control if MS/MSD recoveries are out of control. See also blank spike.
QC Period	Quality control period. An analysis sequence initiated by the injection of BFB, followed by the standard. A QC period is terminated after 12 hours from the injection of BFB.

RF	Response factor. A measure of the response of an analyte compared to its internal standard response. Response factors are determined by standard analysis and are used in the calculation of concentrations of analytes in samples.
RRT	Relative retention time. A measure of the retention time of an analyte compared to the retention time of its internal standard.
RSD or %RSD	Relative standard deviation or percent relative standard deviation. The ratio of the standard deviation of a set of values to the mean of the set of values. A measure of the similarity of the values one to another.
RT	Retention time. The time (in minutes) at which a target analyte elutes from the GC column.
Sequence	A set of samples and standard solutions introduced into an instrument in a chronologically continuous group. See also QC period.
SPCC	System performance check compound. Specified compounds in which the minimum RFs must be met in order to demonstrate that the initial and continuing calibration standards are in control. These compounds are chloromethane, 1,1-dichloroethane, bromoform, chlorobenzene, and 1,1,2,2-tetrachloroethane.

2. Equipment List and Standards

2.1 Chromatographic System

2.1.1 Gas Chromatograph: Hewlett Packard 5890 I or II 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: 30 m x 0.53 mm x 3.0 µm film thickness or 75 m x 0.45 x 2.55 µm film thickness, fused silica capillary column (J&W DB-624 or equivalent).

2.1.4 Purge and Trap: OI Analytical 4460A and 4550 sample concentrators or equivalent.

2.1.5 Autosampler: Dynatech Precision Sampling PTA-30W/S or equivalent.

2.1.6 GC/MS Interface: Hewlett Packard 59913A jet separator assembly or equivalent.

2.1.7 Mass Spectrometer: Hewlett Packard 5970B or equivalent.

2.1.8 Data System: Teknivent.

2.1.9 Trap: OI Corporation, Supelco, or equivalent. Examples are listed below:

1) 1 cm 3% SP-2100/Chromosorb W AW, 7.7 cm Tenax TA, 7.7 cm silica gel 15, 7.7 cm activated charcoal. Supelco Pt No.:2-1139.

2) VOCARB 3000. 10 cm Carbopack C, 6 cm Carboxen 1000, 1 cm Carboxen 1001. Supelco Pt No.: 2-1131.

3) VOCARB 4000. 8.5 cm Carbopack C, 10 cm Carbopack B, 6 cm Carboxen 1000, 1 cm Carboxen 1001. Supelco Pt. No.: 2-1143.

2.1.10 Miscellaneous: Assorted gas-tight calibrated syringes, pipettes, 40 mL vials, caps, septa, sea sand, purge and trap grade methanol, ferrules, pH strips, purge-free DIW, volumetrics, laboratory oven capable of heating glassware up to 100°C, and assorted supplies.

2.2 Standards

2.2.1 Overview of Volatile Standards Preparation

2.2.1.1 All standards are made using a high purity purge and trap grade methanol. Every lot number of methanol is analyzed prior to use to ensure that it is free of volatile organic contaminants. A record of analysis of an aliquot from each batch is stored for a minimum of one year.

2.2.1.2 Two standard logbooks are employed to document all volatile standards. One standard logbook is maintained for stock solutions prepared from neat materials and to record the login of certified standards. The other standard logbook is maintained for working solutions which include solutions procured from commercial sources, as well as those solutions made in the laboratory. All standards are assigned a unique identifier to enable cross-referencing of each individual standard back to the supplier's lot number. In addition, all standards are labeled with the standard concentration, the solvent, date prepared, expiration date, analyst's initials, and the standard reference number. Refer to Laucks' SOP on the traceability, documentation, and preparation of standards.

2.2.1.3 All standards must be identified and quantitated prior to use in the laboratory. This is performed by purchasing certified standard solutions from a commercial supplier that is recognized by the EPA and which are traceable to NIST. In addition, standards for the permanent gases and non-gases are monitored frequently by comparison to the initial calibration curve. Fresh standards should be prepared if the calibration standard(s) fail to meet the criteria specified in SW 846. Standards for gases need to be replaced after one week, or as recommended by the standard manufacturer, unless the acceptability of the standard can be documented. Standards for the non-gases need to be replaced after six months or as recommended by the standard manufacturer, unless the acceptability of the standard can be documented. If the standards meet the criteria for the calibration standard(s) as specified in SW 846, they are considered acceptable.

2.2.1.4 All standard solutions are stored in the VOA freezer at -10°C to -20°C. The stock and secondary dilution standards are stored in flame-sealed amber ampules (with the exception of the internal and surrogate standards). The secondary dilution standards are checked for degradation or evaporation prior to preparing calibration standards. Secondary dilution standards should be replaced after one week, unless the acceptability of the standard can be documented. Pre-mixed certified solutions are stored according to the manufacturers' documented holding time and storage temperature recommendations. Prior to daily and initial calibration standard preparation, the standard solutions are removed from the freezer and allowed to warm to room temperature.

2.2.1.5 Refer to Appendix I for a tabular listing of all standard solutions and their concentrations. The additional appendix IX compounds (which are only analyzed upon request) and their standard concentrations are tabulated in Appendix IV.

2.2.2 Preparation of Internal Standards and Surrogate Standards

2.2.2.1 If starting with a neat material, a measured amount of the neat chemical is placed in a glass volumetric and diluted to volume with methanol, then stoppered. 125 mg of neat material is diluted into 25 mL of methanol. This yields an intermediate solution of 5000 µg/mL.

2.2.2.2 Aliquots of the intermediate solutions are taken from the individual internal and surrogate standards and then combined in a volumetric flask and diluted with methanol to yield a working standard which contains all compounds at a concentration of 250 µg/mL each. This is accomplished by using a gas-tight syringe to transfer each aliquot of 1.25 mL into 25 mL of methanol. A 1 µL aliquot of this solution is injected via the autosampler into 5 mL for waters, low and medium level soils (and 25 mL for low level

waters) of sample, resulting in an internal and surrogate standard concentration of 50 µg/L (or 10 µg/L for low level waters).

Internal Standards	Surrogate Standards
Fluorobenzene	1,2-Dichloroethane-d4
Chlorobenzene-d5	Toluene-d8
1,4-Dichlorobenzene-d4	p-Bromofluorobenzene

2.2.3 Preparation of Matrix Spike Solution

2.2.3.1 At a minimum five volatile analytes are used for spiking. Upon request by the client additional analytes may be used if they are of interest at the site. A certified matrix spiking solution which contains the five spiking analytes is purchased pre-mixed from a commercial supplier at a concentration of 2500 µg/mL each. This solution is then diluted in methanol to a concentration of 200 µg/mL.

Matrix Spike Solution	Source*	Part Number*
1,1-Dichloroethene	Restek	30005
Benzene		
Trichloroethene		
Toluene		
Chlorobenzene		
* or equivalent		

2.2.3.2 A 12.5 µL (2.5 µL for low level waters) aliquot of this solution is spiked into 50 mL of sample, resulting in a spiked concentration of 50 µg/L (or 10 µg/L for low level waters) for each analyte.

Working Concentration	Aliquot	Final Volume	Concentration
Soils, 5 mL Water			
200 µg/mL	12.5 µL	50 mL PFW	50 µg/L
Medium Level Soils			
200 µg/mL	12.5 µL	50 mL PFW	(50 µg/L extract) 5000 µg/L sample
25 mL Water			
200 µg/mL	2.5 µL	50 mL PFW	10 µg/L

2.2.4 Preparation of p-Bromofluorobenzene (BFB) Solution

2.2.4.1 The preparation of BFB is similar to that of the internal standards and surrogate standards. Refer to Section 2.2.2 for details.

2.2.4.2 A solution of 25 ng/L is prepared and stored in flame-sealed amber ampules.

2.2.5 Preparation of Initial Calibration Standards

2.2.5.1 Certified sets of stock solutions which contain all target analytes are purchased pre-mixed from a commercial supplier at a concentration of 2000 µg/mL each. If additional target analytes are requested, these analytes may be ordered separately, either in solution or as a neat material.

2.2.5.2 These solutions are then diluted in methanol to generate standards at concentrations of 200 µg/mL. This solution is stored in flame-sealed amber ampules.

2.2.5.3 Standards are stored in the VOA freezer at -10°C to -20°C. They are removed prior to making up the initial calibration standards and are allowed to warm to room temperature.

2.2.5.4 The initial calibration standards range from 1 µg/L to 50 µg/L for low level waters and 3 µg/L to 200 µg/L for soils, and 5 mL water samples. 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 require 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 containing PFW. A 40 mL aliquot is immediately poured into a VOA vial (or a 5 mL aliquot for the low level soils is placed in a soil vessel) and placed on the autosampler. Each standard concentration is prepared just prior to analysis and any remaining standard solution is disposed of once the analytical run is initiated.

Solution	Source*	Part Number*
VOC Mix 1	Supelco	4-8775
VOC Mix 2	Supelco	4-8777
VOC Mix 3	Supelco	4-8779
VOC Mix 4	Supelco	4-8786
VOC Mix 5	Supelco	4-8797
VOC Mix 6	Supelco	4-8799
TCL Mix 1	Supelco	4-8949
Carbon Disulfide	Supelco	4-0363

* or equivalent

Low Level Water (25 mL)

Working Solution	Aliquot non-ketones	Aliquot ketones	Final Volume	Concentrations non-ketones	Concentrations ketones
200 µg/mL	1.0 µL	2.0 µL	200 mL	1 µg/L	2 µg/L
200 µg/mL	2.5 µL	5 µL	100 mL	5 µg/L	10 µg/L
200 µg/mL	5.0 µL	10 µL	100 mL	10 µg/L	20 µg/L
200 µg/mL	6.2 µL	12.5 µL	50 mL	25 µg/L	50 µg/L
200 µg/mL	12.5 µL	25 µL	50 mL	50 µg/L	100 µg/L

Low Level Soil

Working Solution	Aliquot non-ketones	Aliquot ketones	Final Volume	Concentrations non-ketones	Concentrations ketones
200 µg/mL	1.5 µL	2.5 µL	100 mL/50 mL	3 µg/L	10 µg/L
200 µg/mL	2.5 µL	5 µL	50 mL	10 µg/L	20 µg/L
200 µg/mL	6.25 µL	6.25 µL	25 mL	50 µg/L	50 µg/L
200 µg/mL	5.0 µL	5.0 µL	10 mL	100 µg/L	100 µg/L
200 µg/mL	10.0 µL	10.0 µL	10 mL	200 µg/L	200 µg/L

5 mL Water And Medium Level Soil

Working Solution	Aliquot non-ketones	Aliquot ketones	Final Volume	Concentrations non-ketones	Concentrations ketones
200 µg/mL	1.5 µL	2.5 µL	100 mL/50 mL	3 µg/L	10 µg/L
200 µg/mL	2.5 µL	2.5 µL	50 mL	10 µg/L	20 µg/L
200 µg/mL	12.5 µL	12.5 µL	50 mL	50 µg/L	50 µg/L
200 µg/mL	25 µL	25 µL	50 mL	100 µg/L	100 µg/L
200 µg/mL	50 µL	50 µL	50 mL	200 µg/L	200 µg/L

Appendix IX Analytes (25 mL)

Working Solution	Aliquot	Final Volume	Concentrations
200 µg/mL	2.5 µL	50 mL	10 µg/L
200 µg/mL	5 µL	50 mL	20 µg/L
200 µg/mL	12.5 µL	50 mL	50 µg/L
200 µg/mL	25 µL	50 mL	100 µg/L
200 µg/mL	50 µL	50 mL	200 µg/L

2.2.6 Preparation of a Daily Calibration Standard

2.2.6.1 The concentration of the continuing calibration standard at a level of 10 µg/L for low level waters, 50 µg/L for low and medium level soils and 5 mL waters, is also the mid-point of the initial calibration curve. This standard is always prepared just prior to

analysis by transferring an aliquot of each of the 200 µg/mL multi-component working solutions into a volumetric containing PFW.

3. Safety precautions & Waste disposal

3.1 Routine Safety Precautions

3.1.1 All standards and samples should be handled as if they are hazardous substances.

3.1.2 Refer to the instrument manufacturer's manual for routine instrument precautions.

3.1.3 Routine precautions include an awareness of the moving parts on the instrument you're using. These parts are often charged with power from an electrical component or with high pressure gas and have the potential to do harm if not used properly.

3.1.4 Electrical shock - All instruments present the possibility of electrical shock. The operator should take all precautions including ensuring that all instruments are operated with fully grounded power outlets, turning off the instrument and disconnecting the instrument from the electrical power supply before working on any electrical components, etc.

3.1.5 Flammable solvents such as methanol are stored in the appropriate solvent locker located outside the volatile laboratory.

3.1.6 Many analytes are known or suspected carcinogens. Analysts should take the proper precautions such as wearing gloves when working with suspect samples or high level standards or solvents. Additionally, a respirator should be worn and a fume hood utilized for extremely hazardous compounds.

3.2 Waste disposal

3.2.1 Waste solvents and expired standards are disposed of in the appropriate waste solvent container located in the prep area under the hood.

3.2.2 Solid sample matrices are disposed of in appropriate drums labeled for this specific purpose where they are routinely picked up by an approved agency for final disposal.

4. Operation procedures

4.1 Analytical Conditions

GC Parameters

Injection Port Temperature	: 200°C
GC/MS Interface Temperature	: 250°C
Initial GC Temperature	: 30°C
Initial Hold Time	: 4 min
Ramp Rate	: 4°C/min
Final Temperature	: 140°C
Final Time	: 4 min
Carrier Gas Flow	: 15 mL/min
Column Head Pressure	: 9 psi

Purge and Trap Parameters

Purge Time	: 11 min
Desorb Time	: 1 min
Bake Time	: 16 min
Desorb Preheat Temperature	: 20°C
Desorb Temperature	: 180°C
Purge Flow	: 40 mL/min
Purge Temperature	: Ambient (40°C for soils)
Desorb Flow Rate	: 15 mL/min

Mass Spectrometer Parameters

Electron Energy	: 70 volts
Mass Range	: 35 to 300 amu
Scan Time	: 1.4 sec/scan
Scan Start Time	: 0.1 min

4.2 Method Detection Limit Study

4.2.1 Prior to the analysis of any samples, it is necessary to establish method detection limits. This procedure is fully described in the Laucks SOP for the determination of method detection limits. It involves the analysis of 7 replicate samples spiked at a concentration near the anticipated method detection limit. A Student's T-test is then applied to these measured values to calculate the MDL.

4.3 Method Validation

4.3.1 Prior to the analysis of any samples, it is necessary to validate the method. A method validation study is performed in a similar manner to an MDL study with the exception that a minimum of 4 replicates are required and the concentration levels are typically 10-50 times the MDL. In the absence of criteria for the initial demonstration of proficiency, the limits of 70%- 130% can be used.

4.4 Instrument Tuning

4.4.1 Prior to the analysis of any samples, blanks, or calibration standards, the instrument must demonstrate mass calibration and resolution by meeting the established tuning criteria for BFB.

4.4.2 FC-43 (PFTBA) is used as a mass calibrant. The following ratios are suggested in order to meet the tuning criteria for BFB:

Ion	% of ion 69	Isotope	Isotope Abundances
69	100%	70	0.5-1.5%
131	25-40%	132	2.0-4.0%
219	25-40%	220	2.5-5.0%
414	1-3%		

4.4.3 In addition to the tabulated abundances listed above, other criteria should be reviewed at this time.

1) Ion peak widths: The appropriate range is between 0.5 and 0.6 amu. Amu widths beyond this range can lead to loss of minor isotopes, while insufficient peak widths will result in decreased sensitivity.

2) Leak check: The abundances of ions 18, 28, and 32 (water, nitrogen and oxygen, respectively) should be less than 5 percent relative to 69. A level greater than 5 percent is indicative of a leak in the system.

4.4.4 Following the system tuning using FC-43, the GC/MS is then calibrated with BFB. A 2 µL aliquot (1 µL for low level waters) of 25 ng/µL of BFB is injected directly into the GC resulting in a concentration of 50 ng (25 ng for low level waters).

4.4.5 The ion abundances and ratios are checked against the criteria detailed below **or** alternatively, other criteria may be used (e.g., CLP, 524.2). If BFB meets the tuning criteria, then the system is determined to be calibrated. If BFB does not meet the tuning criteria, the GC/MS is re-tuned with FC-43 as described above.

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 Analyze standard solutions using a minimum of 5 different concentration levels. The lowest concentration should establish the reporting limit. The highest concentration should define the upper usable working range of the detector. Criteria for evaluating these standards are detailed in Section 6.

4.6 Continuing Calibration Verification

4.6.1 A CCV is analyzed once the BFB tuning criteria have been met and the instrument has been calibrated. Criteria for evaluating a CCV standard are detailed in Section 6.

4.7 Instrument Blank

4.7.1 The analysis of an instrument blank (IBLK) is performed prior to the injection of BFB. This analysis ensures that there are no volatile organic contaminants in either the purge and trap sample concentrator or in the autosampler.

4.7.2 Any sample that demonstrates concentrations of target analytes high enough to saturate the detector must be followed by at least one IBLK, also known as a saturation blank. This IBLK analysis is performed to prevent any possible carry-over into the subsequent sample analysis. Evaluation criteria follow the same guidelines set forth for the analysis of a method blank.

4.8 Method Blank

4.8.1 Immediately following the analysis of the CCV and prior to sample analysis, a method blank must be analyzed. The analysis of the method blank demonstrates that the instrument is free of volatile organic contaminants and ensures that the reagent water is also free of contaminants. Refer to Section 6 for method blank acceptance criteria.

4.9 Sample Analysis

4.9.1 Samples (and associated QC) may be analyzed for up to 12 hours from the injection of BFB. The samples are analyzed subsequent to the calibration standard and method blank analyses. However, no more than twenty sample analyses are performed in a 12 hour period. Once all criteria for BFB, the calibration standard, and method blank have been met, the sample analyses are performed. The aqueous and medium level soil samples are analyzed against an aqueous (non-heated purge) calibration curve. The low level soils are analyzed against a calibration curve employing a heated purge temperature of 40°C.

4.10 Analysis sequence

IBLK (optional)
BFB
CCV
Method blank
Samples and QC

4.11 Sample Preparation

4.11.1 The samples are removed from the VOA refrigerator and are allowed to warm to room temperature. Aqueous samples which are received in 40 mL vials and low level soil

samples containing the pre-weighed sample aliquot in sodium bisulfate are placed onto the autosampler carousel. See section 4.11.5 for the preparation of medium soils. The autosampler transfers a 5 mL, or 25 mL aliquot of sample (or an aliquot of PFW for the soils) into the autosampler syringe, where it injects 1 µL of the internal standard and surrogate standard solution.

4.11.2 The pH of all water and low level soil samples is measured subsequent to analysis in order to determine if they were preserved adequately without disturbing the integrity of the sample. The pH values are noted in the instrument logbook.

4.11.3 If the concentration of any target analyte exceeds the initial calibration range, the sample must be diluted and reanalyzed.

4.11.4 If aqueous samples require dilutions, they are diluted by one of two methods listed:

1) the samples are diluted with PFW in a volumetric where they are transferred to 40 mL vials and placed onto the autosampler, or

2) the undiluted samples are placed on the autosampler, where they are diluted with PFW by the autosampler. Refer to the instrumentation manuals for the complete operation of the autosampler.

4.11.5 Medium Soils

4.11.6 The soil sample is gently mixed (no supernatant liquid is removed) and a 5 gram aliquot (weighed out to the nearest 0.1 g) is transferred to a tared 15 mL vial. 10.0 mL of methanol is added to the vial. The vial is sealed, then shaken for two minutes. A 1 mL aliquot is transferred to a GC vial for storage in the dark at 4°C (±2°C). A 100 µL sample aliquot per 5 mL of PFW is placed onto the autosampler carousel. The autosampler transfers a volume of 5 mL of this sample into the autosampler syringe, where it injects 1 µL of the internal standard and surrogate solution.

4.12 Qualitative Identification

4.12.1 Two criteria must be satisfied to verify the identification of target compounds:

1) elution of the sample component within 0.50 minutes of the standard component and,

2) appropriate correspondence between the sample mass spectrum and the standard mass spectrum.

a) The three ions of greatest relative intensity, or any ions over 30% relative intensity which are present in the standard mass spectrum must be present in the sample spectrum.

b) The relative intensities of ions must be within 30% between the standard and sample spectrum.

d) The emphasis of spectral review is on the three major ions. However, if a target compound cannot be verified by all the criteria listed above, but if, in the technical judgment of the analyst, the identification is correct, then the analyte will be quantitated, and reported. Compounds meeting the identification criteria must be reported with their spectra.

4.12.2 A library search may be performed for the purpose of identifying non-target analytes also referred to as Tentatively Identified Compounds (TICs). 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.

4.13 Compound Quantification

4.13.1 Target compound concentrations are calculated using the following equations:

<u>Aqueous samples:</u>

$$\text{Conc } (\mu g / L) = \frac{A_x \times I_s \times Df}{A_{is} \times ARF \times V_o}$$

where:

A_x = Area of the characteristic ion (EICP) of the analyte.
A_{is} = Area of the characteristic ion (EICP) of the internal standard.
I_s = Amount of the internal standard (ng).
ARF = Average RF from the ambient temperature purge of the initial calibration.
V_o = Volume of water purged (mL).
Df = Dilution factor. Assume 1.0 for no dilution.

Low soil (on a dry-weight basis)

$$\text{Conc } (\mu g / kg) = \frac{A_x \times I_s}{A_{is} \times ARF \times W_s \times D}$$

where:

A_x = Area of the characteristic ion (EICP) of the analyte.
A_{is} = Area of the characteristic ion (EICP) of the internal standard.
I_s = Amount of the internal standard (ng).
ARF = Average response factor from the heated purge of the initial calibration.
W_s = Weight of sample added to purge vessel (g).

$$D = \frac{100 - \%\text{Moisture}}{100}$$

Medium soil (on a dry weight basis)

$$\text{Conc } (\mu g / kg) = \frac{A_x \times I_s \times V_t \times 1000 \times Df}{A_{is} \times ARF \times V_a \times W_s \times D}$$

where:

A_x = Area of the characteristic ion (EICP) of the analyte.
A_{is} = Area of the characteristic ion (EICP) of the internal standard.
I_s = Amount of the internal standard (ng).
ARF = Average RF from the ambient temperature purge of the calibration standard.
V_t = Total volume of the methanol extract in milliliters (10 mL).
V_a = Volume of the methanol extract in microliters added to the reagent water for purging.
W_s = Weight of sample added to purge vessel (g).
Df = Dilution factor. Assume 1.0 for no dilution.

5. Reports

5.1 Data Packet Organization

5.1.1 See Appendix II for a check list detailing data packet organization.

5.2 Quality Control Reports

5.2.1 All results for quality control tests are entered into the lab database. Printouts of all data entered must be included in the data packet. The routine minimum is a method blank report, a blank spike report, and a MS/MSD report.

5.3 Control Limits

5.3.1 The laboratory has a computerized database which is used to generate control limits for various analyses, matrices, and analytes. The control limits define the range in which 99% of all values produced by a system should lie. Because the control limits are updated periodically, the current control limits for QC analyses have not been defined in this SOP. A copy of all current control limits are located in Laucks' Control Limits Database.

5.4 Data Qualifying Flags

5.4.1 Sample report results are qualified with data qualifying flags. These flags have the following definitions:

Code	Definition
U	The analyte of interest was not detected, to the limit of detection indicated.
B	The analyte of interest was detected in the method blank associated with the sample, as well as in the sample itself. The B flag is applied without regard to the relative concentrations detected in the blank and sample.
J	The analyte of interest was detected below the practical quantitation limit. This value should be regarded as an estimate.
D	The value reported is derived from the analysis of a diluted sample or sample extract.
E	The value reported is based on a sample or sample extract in which the target analyte concentration exceeded the calibration range. The value reported should be considered an estimate.
N	Used to denoted the presumptive evidence of a target analyte. This flag is used when it is not possible to confirm the presence of a compound by the strict guidelines for mass spectral interpretation, but in the technical judgment of the analyst, this compound is present.

6. Quality Control

6.1 Initial Calibration

6.1.1 Criteria

6.1.1.1 Initial calibration data are evaluated using %RSD and the relative response factors.

RFs are calculated using the equation:

$$RF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_s}$$

where:

A_x = Response of target analyte
A_{is} = Response of internal standard
C_x = Amount of target analyte (ng/mL).
C_{is} = Amount of internal standard (ng/mL).

%RSDs are calculated using the equation:

$$\%RSD = \frac{SD_i}{RF_i} \times 100$$

where:

Sd_i = Standard deviation of the initial calibration points
RF_i = Average RF from initial calibration

6.1.2 The System Performance Check Compounds (SPCCs) are checked to determine that the minimum average relative response factors tabulated below are met, and that the maximum RSD for each Calibration Check Compound (CCC) is < 30%. The RSD for all other target analytes *should* be ≤ 15%. If the RSD of any target analyte is greater than 15%, but < 30%, additional calibration options may be used. Alternatively, if the mean of the RSD values for all analytes in the calibration is < 15%, the initial calibration is considered acceptable.

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%

6.1.2.3 For Navy samples, an additional criterion of a minimum RRF of 0.005 for all other reported analytes (with the exception 25 mL purges) has been implemented.

6.1.3 Corrective action

6.1.3.1 If the criteria are not met, additional standards may be analyzed or appropriate instrument maintenance and analysis of new standards should be performed.

6.1.4 Documentation

6.1.4.1 Copies of the initial calibration standards and the calculated RRFs and RSDs are stored with the raw data. If criteria are not met, reanalysis of additional standard(s) are noted in the instrument run log.

6.2 Continuing Calibration Verification

6.2.1 Criteria

6.2.1.1 At the beginning of each 12 hour QC period, a CCV standard is analyzed. The RRF for each compound is calculated and the percent difference is calculated as follows:

$$\%D = \frac{RF_i - RF_c}{RF_i} \times 100$$

where:

RF_i = Average RF from Initial Calibration
RF_c = RF from CCV standard

6.2.1.2 The minimum RRFs for the SPCCs must meet the criteria applied in the initial calibration detailed above. The %D for the CCCs cannot exceed the 20% CCV criteria as tabulated below.

CCC Compounds	% D Limit
1,1-Dichloroethene	<20%
Chloroform	<20%
1,2-Dichloropropane	<20%
Toluene	<20%
Ethylbenzene	<20%
Vinyl chloride	<20%

6.2.1.3 For Navy samples, additional criteria of a maximum %D of 40% and a minimum RF of 0.005 for all other reported analytes (with the exception of 25 mL purges)has been implemented.

6.2.2 Corrective action

6.2.2.1 Check calculations, check standard solutions or perform instrument maintenance. Reanalyze the standard.

6.2.3 Documentation

6.2.3.1 Copies of the continuing calibration standards and the calculated RFs and %Ds are stored with the raw data. If criteria are not met, reanalysis of additional standard(s) are noted in the instrument run log.

6.3 Instrument Blank

6.3.1 Criteria

6.3.1.1 The criteria used are the same as those used for the method blank control limits.

6.3.2 Corrective action

6.3.2.1 If the initial IBLK contains measurable levels of target analytes above 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 if free of contaminants, then instrument maintenance such as cleaning the transfer lines or replacing the trap may be required.

6.3.3 Documentation

6.3.3.1 Copies of instrument blank analyses are stored with the sample analyses raw data. Re-analysis of the method blank and/or maintenance performed is documented in the instrument run log.

6.4 Method Blanks

6.4.1 Criteria

6.4.1.1 A method blank is used to verify contamination free reagents and apparatus. A method blank is analyzed following every CCV and prior to sample analysis. The results of the method blank should be:

6.4.1.2 Less than the laboratory's MDL, or less than the level of acceptable contamination specified in the approved quality assurance plan. Laucks criteria for a method blank is less than the RL for the analytes with the exception of methylene chloride and acetone which are allowed up to 5 times the RL.

6.4.1.3 Less than 5% of the regulatory limit associated with an analyte.

6.4.1.4 Or less than 5% of the sample result for the same analyte, whichever is greater.

6.4.2 Corrective action

6.4.2.1 Corrective action in the form of reanalysis of the method blank prior to sample analysis is performed when target analytes are present. However, the detection of non-target

analytes does not require corrective action if they are common laboratory solvents such as 1,1,2-trichloro-1,2,2-trifluoroethane (freon).

6.4.2.2 Out-of-control surrogate recoveries in the method blank require reanalysis of the method blank.

6.4.3 Documentation

6.4.3.1 Copies of all method blank analyses are stored with the sample analyses raw data. In addition, the raw data for the method blank analyses are stored in the laboratory for an extended period of time. The results for the method blank analyses are reported electronically via the laboratory's LIMS database. If criteria are not met, reanalysis of additional blank(s) are noted in the instrument run log.

6.5 Blank Spikes

6.5.1 Criteria

6.5.1.1 A blank spike follows the same protocol as a matrix spike analysis except that the spiking solution is added to an aliquot of PFW (or sand for soil samples) instead of an actual sample. A method blank with added analytes is a blank spike. A blank spike is the same as a QC check standard.

6.5.2 Corrective action

6.5.2.1 The blank spike is used to determine whether a method is in control during sample preparation and analysis. Sample reanalysis would be triggered by an out of control blank spike only if the sample surrogate recoveries and MS/MSD spike recoveries indicated sample processing errors.

6.5.3 Documentation

6.5.3.1 The raw data for the blank spike analyses are stored in the laboratory for an extended period of time. The results for the blank spike analyses are reported electronically via the laboratory's LIMS database. If criteria are not met, reanalysis of additional blank spike(s) are noted in the instrument run log.

6.6 Matrix Spike

6.6.1 Criteria

6.6.1.1 A sample is chosen at random (unless specified by a client) from the samples to be analyzed, and an aliquot of spiking solution is added to this sample prior to analysis. A matrix spike analysis is performed with each analytical batch, up to a maximum of 20 samples. The matrix spike sample is used to evaluate the matrix effect of the sample upon recovery of the analytes. The recovery of spike analytes is calculated as follows:

$$\text{Recovery} = \frac{SS - S}{SpikedAmount} \times 100$$

where:

SS = concentration in spiked sample
S = native concentration in unspiked sample

6.6.2 Corrective action

6.6.2.1 Samples with spike recoveries outside control limits will be reviewed for possible corrective action. Corrective action may involve recalculation and/or reanalysis. This process should also include evaluation of the recovery of surrogate compounds in the MS sample and recovery of matrix spiking compounds from the blank spike analysis. In all cases a narrative explanation of the condition is required to detail the corrective actions taken.

6.6.3 Documentation

6.6.3.1 The raw data for the matrix spike analyses are stored with the sample analyses raw data. The results for the matrix spike analyses are reported electronically via the laboratory's LIMS database.

6.7 Matrix Spike Duplicate

6.7.1 Criteria

6.7.1.1 A matrix spike duplicate analysis is performed with each analytical batch, up to a maximum of 20 samples. The compound recovery criteria are identical to those for the matrix spike sample. In addition, the matrix spike duplicate is used to measure method precision. This is done by computing the relative percent difference (RPD) between the matrix spike and matrix spike duplicate recovery values. This calculation is as follows:

$$RPD = \frac{S1 - S2}{(S1 + S2) \backslash 2} \times 100$$

where:

S1 = measured concentration for MS sample
S2 = measured concentration for MSD sample

6.7.2 Corrective action

6.7.2.1 If a trend in out-of-control RPD values is observed, the method used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable reproducibility.

6.7.3 Documentation

6.7.3.1 The raw data for the matrix spike duplicate analyses are stored with the sample analyses raw data. The results for the matrix spike duplicate analyses are reported electronically via the laboratory's LIMS database. Any out-of-control results are documented in the LIMS database and/or analytical narrative.

6.8 Surrogate Recovery

6.8.1 Criteria

6.8.1.1 Surrogates are chemically similar compounds added to every sample, method blank, and QC sample prior to sample processing. They are used to monitor potential sample processing errors and matrix effects. Surrogate compound recoveries are calculated as follow:

$$\text{Recovery} = \frac{S_m}{S_a} \times 100$$

where:

S_m = concentration of surrogate measured in sample
S_a = concentration of surrogate added

6.8.2 Corrective Action

6.8.2.1 Check calculations for possible error. Reanalysis is required for all surrogate recoveries which exceed the established control limits.

6.8.2.2 Out-of-control surrogate recoveries in the method blank require that the method blank be reanalyzed. Sample analyses are not performed until all criteria are met in the method blank analysis. Sample and QC analyses require reanalysis for out-of-control surrogate recoveries. If the sample reanalyses also demonstrate out-of-control surrogate recoveries due to matrix interference, this corrective action is discussed in the narrative.

6.8.3 Documentation

6.8.3.1 The surrogate recoveries for all samples, blanks, and QC analyses are reported electronically via the laboratory's LIMS database. If criteria are not met, reanalysis of for out-of-control surrogate recoveries are designated by an "RE" in the instrument run log.

7. References

1) Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), US EPA Office of Solid Waste and Emergency Response, - Method 8260B, 3rd edition, 2nd revision, December 1996.

2) Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), US EPA Office of Solid Waste and Emergency Response, - Method 8000B, 3rd edition, 2nd revision, December 1996.

3) Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), US EPA Office of Solid Waste and Emergency Response, - Method 5000B, 3rd edition, revision 0, December 1996.

4) Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), US EPA Office of Solid Waste and Emergency Response, - Method 5030B, 3rd edition. 2nd revision, December 1996.

5) Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), US EPA Office of Solid Waste and Emergency Response, - Method 5035, 3rd edition, revision 0, December 1996.

APPENDIX I

Initial Calibration Concentrations For Low Level (25 mL) Waters (µg/L)

Compound	STD1	STD2	STD3	STD4	STD5
Dichlorodifluoromethane	1	5	10	25	50
Chloromethane	1	5	10	25	50
Vinyl Chloride	1	5	10	25	50
Bromomethane	1	5	10	25	50
Chloroethane	1	5	10	25	50
Trichlorofluoromethane	1	5	10	25	50
1,1-Dichloroethene	1	5	10	25	50
Acetone	5	10	20	50	100
Carbon disulfide	1	5	10	25	50
Methylene chloride	1	5	10	25	50
trans-1,2-Dichloroethene	1	5	10	25	50
1,1-Dichloroethane	1	5	10	25	50
Vinyl acetate	1	5	10	25	50
2,2-Dichloropropane	1	5	10	25	50
cis-1,2-Dichloroethene	1	5	10	25	50
2-Butanone	5	10	20	50	100
Bromochloromethane	1	5	10	25	50
Chloroform	1	5	10	25	50
1,1,1-Trichloroethane	1	5	10	25	50
Carbon tetrachloride	1	5	10	25	50
1,1-Dichloropropene	1	5	10	25	50
Benzene	1	5	10	25	50
1,2-Dichloroethane	1	5	10	25	50
Trichloroethene	1	5	10	25	50
1,2-Dichloropropane	1	5	10	25	50
Dibromomethane	1	5	10	25	50
Bromodichloromethane	1	5	10	25	50
cis-1,3-Dichloropropene	1	5	10	25	50
4-Methyl-2-pentanone	5	10	20	50	100
Toluene	1	5	10	25	50
trans-1,3-Dichloropropene	1	5	10	25	50

Compound	STD1	STD2	STD3	STD4	STD5
1,1,2-Trichloroethane	1	5	10	25	50
Tetrachloroethene	1	5	10	25	50
1,3-Dichloropropane	1	5	10	25	50
2-Hexanone	5	10	20	50	100
Chlorodibromomethane	1	5	10	25	50
1,2-Dibromoethane	1	5	10	25	50
Chlorobenzene	1	5	10	25	50
1,1,1,2-Tetrachloroethane	1	5	10	25	50
Ethylbenzene	1	5	10	25	50
m,p-Xylene	2	10	20	50	100
o-Xylene	1	5	10	25	50
Styrene	1	5	10	25	50
Bromoform	1	5	10	25	50
Isopropylbenzene	1	5	10	25	50
Bromobenzene	1	5	10	25	50
1,2,3-Trichloropropane	1	5	10	25	50
1,1,2,2-Tetrachloroethane	1	5	10	25	50
n-Propylbenzene	1	5	10	25	50
2-Chlorotoluene	1	5	10	25	50
4-Chlorotoluene	1	5	10	25	50
1,3,5-Trimethylbenzene	1	5	10	25	50
tert-Butylbenzene	1	5	10	25	50
1,2,4-Trimethylbenzene	1	5	10	25	50
sec-Butylbenzene	1	5	10	25	50
1,3-Dichlorobenzene	1	5	10	25	50
1,4-Dichlorobenzene	1	5	10	25	50
p-Isopropyltoluene	1	5	10	25	50
1,2-Dichlorobenzene	1	5	10	25	50
n-Butylbenzene	1	5	10	25	50
1,2-Dibromo-3-chloropropane	1	5	10	25	50
1,2,4-Trichlorobenzene	1	5	10	25	50
Naphthalene	1	5	10	25	50
Hexachlorobutadiene	1	5	10	25	50
1,2,3-Trichlorobenzene	1	5	10	25	50

Initial Calibration Concentrations Of Routine Target Analytes For Low And Medium Level Soils & 5 mL Waters

Compound (µg/L)	STD1	STD2	STD3	STD4	STD5
Dichlorodifluoromethane	3	10	50	100	200
Chloromethane	3	10	50	100	200
Vinyl Chloride	3	10	50	100	200
Bromomethane	3	10	50	100	200
Chloroethane	3	10	50	100	200
Trichlorofluoromethane	3	10	50	100	200
1,1-Dichloroethene	3	10	50	100	200
Acetone	10	20	50	100	200
Carbon disulfide	3	10	50	100	200
Methylene chloride	3	10	50	100	200
trans-1,2-Dichloroethene	3	10	50	100	200
1,1-Dichloroethane	3	10	50	100	200
cis-1,2-Dichloroethene	3	10	50	100	200
2-Butanone	10	20	50	100	200
Chloroform	3	10	50	100	200
1,1,1-Trichloroethane	3	10	50	100	200
Carbon tetrachloride	3	10	50	100	200
Benzene	3	10	50	100	200
1,2-Dichloroethane	3	10	50	100	200
Trichloroethene	3	10	50	100	200
1,2-Dichloropropane	3	10	50	100	200
Bromodichloromethane	3	10	50	100	200
cis-1,3-Dichloropropene	3	10	50	100	200
4-Methyl-2-pentanone	10	20	50	100	200
Toluene	3	10	50	100	200
trans-1,3-Dichloropropene	3	10	50	100	200
1,1,2-Trichloroethane	3	10	50	100	200
Tetrachloroethene	3	10	50	100	200
2-Hexanone	10	20	50	100	200
Chlorodibromomethane	3	10	50	100	200
Chlorobenzene	3	10	50	100	200
Ethylbenzene	3	10	50	100	200
m,p-Xylene	6	20	100	200	400
o-Xylene	3	10	50	100	200
Styrene	3	10	50	100	200
Bromoform	3	10	50	100	200

Compound (µg/L)	STD1	STD2	STD3	STD4	STD5
1,1,2,2-Tetrachloroethane	3	10	50	100	200

Initial Calibration Concentrations Of
Additional Analytes For Low And Medium Level Soils & 5 mL Waters

Compound (µg/L)	STD1	STD2	STD3	STD4	STD5
1,1,1,2-Tetrachloroethane	3	10	50	100	200
2,2-Dichloropropane	3	10	50	100	200
Bromochloromethane	3	10	50	100	200
1,1-Dichloropropene	3	10	50	100	200
1,2-Dibromoethane	3	10	50	100	200
Dibromomethane	3	10	50	100	200
1,3-Dichloropropane	3	10	50	100	200
Isopropylbenzene	3	10	50	100	200
Bromobenzene	3	10	50	100	200
1,2,3-Trichloropropane	3	10	50	100	200
n-Propylbenzene	3	10	50	100	200
2-Chlorotoluene	3	10	50	100	200
4-Chlorotoluene	3	10	50	100	200
1,3,5-Trimethylbenzene	3	10	50	100	200
tert-Butylbenzene	3	10	50	100	200
1,2,4-Trimethylbenzene	3	10	50	100	200
sec-Butylbenzene	3	10	50	100	200
1,3-Dichlorobenzene	3	10	50	100	200
1,4-Dichlorobenzene	3	10	50	100	200
p-Isopropyltoluene	3	10	50	100	200
1,2-Dichlorobenzene	3	10	50	100	200
n-Butylbenzene	3	10	50	100	200
1,2-Dibromo-3-chloropropane	3	10	50	100	200
1,2,4-Trichlorobenzene	3	10	50	100	200
Naphthalene	3	10	50	100	200
Hexachlorobutadiene	3	10	50	100	200
1,2,3-Trichlorobenzene	3	10	50	100	200
Iodomethane	3	10	50	100	200
trans-1,4-Dichloro-2-butene	3	10	50	100	200

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
   - Chromatogram normalized to nonsolvent peak
   - Quantitation report
   - Spectra

3. Standards Data
   - Initial calibration form
   - Chromatograms
   - Quantitation reports

   - CCV form
   - Chromatograms
   - Quantitation reports

4. Raw QC
   - BFB tune spectrum, chromatogram, and tabular listing
   - Method blank results, TICs, chromatogram, quantitation report, and spectra
   - MS/MSD results, chromatogram, quantitation report, spectra and control charts

5. Bench Sheets
   - Copy of instrument logbook, copy of COC, copy of holding blank analysis

6. Narrative.

7. Electronic Disk Deliverables (EDD) when requested

8. Control charts

APPENDIX III

Determination of Vinyl Chloride by Selected Ion Monitoring (SIM)

Analytical Conditions:

GC Parameters

Injection Port Temperature	: 200°C
GC/MS Interface Temperature	: 250°C
Initial GC Temperature	: 30°C
Initial Hold Time	: 0 min
Ramp Rate	: 6°C/min
Final Temperature	: 105°C
Final Time	: 0 min
Carrier Gas Flow	: 15 mL/min
Column Head Pressure	: 9 psi

Purge and Trap Parameters

Purge Time	: 11 min
Desorb Time	: 1 min
Bake Time	: 16 min
Desorb Preheat Temperature	: 20°C
Desorb Temperature	: 180°C
Purge Flow	: 40 mL/min
Purge Temperature	: Ambient
Desorb Flow Rate	: 15 mL/min

Mass Spectrometer Parameters

Electron Energy	: 70 volts
Acquisition Start Time	: 0.1 min

Group 1:

M/Z	Dwell Time (milliseconds)
61	50
62	400
63	50
64	50

Group 2:

M/Z	Dwell Time (milliseconds)
49	50
51	50
128	100
129	50

Group 3:

M/Z	Dwell Time (milliseconds)
65	50
67	50
102	50
104	50

Standards:

Internal Standard: Bromochloromethane
Surrogate: 1,2-Dichloroethane-d4

APPENDIX IV

Additional Appendix IX Compounds And Their Initial Concentrations For Low Level (25mL) Waters

Compound* (µg/L)	STD1	STD2	STD3	STD4	STD5
Acrolein	10	20	50	100	200
Bromoethane	10	20	50	100	200
Allyl chloride	10	20	50	100	200
Acrylonitrile	10	20	50	100	200
Chloroprene	10	20	50	100	200
Methacrylonitrile	10	20	50	100	200
Ethyl methacrylate	10	20	50	100	200
Methyl methacrylate	10	20	50	100	200
trans-1,4-Dichloro-2-butene	10	20	50	100	200
2-Chloroethylvinyl ether	10	20	50	100	200

* The calibration range defines the reporting range for these compounds. However, the reporting limits may change for individual projects, based on MDLs, mass spectral confirmation, or project-specific requirements.

APPENDIX V

Method 8260 Requirements and Corrective Actions

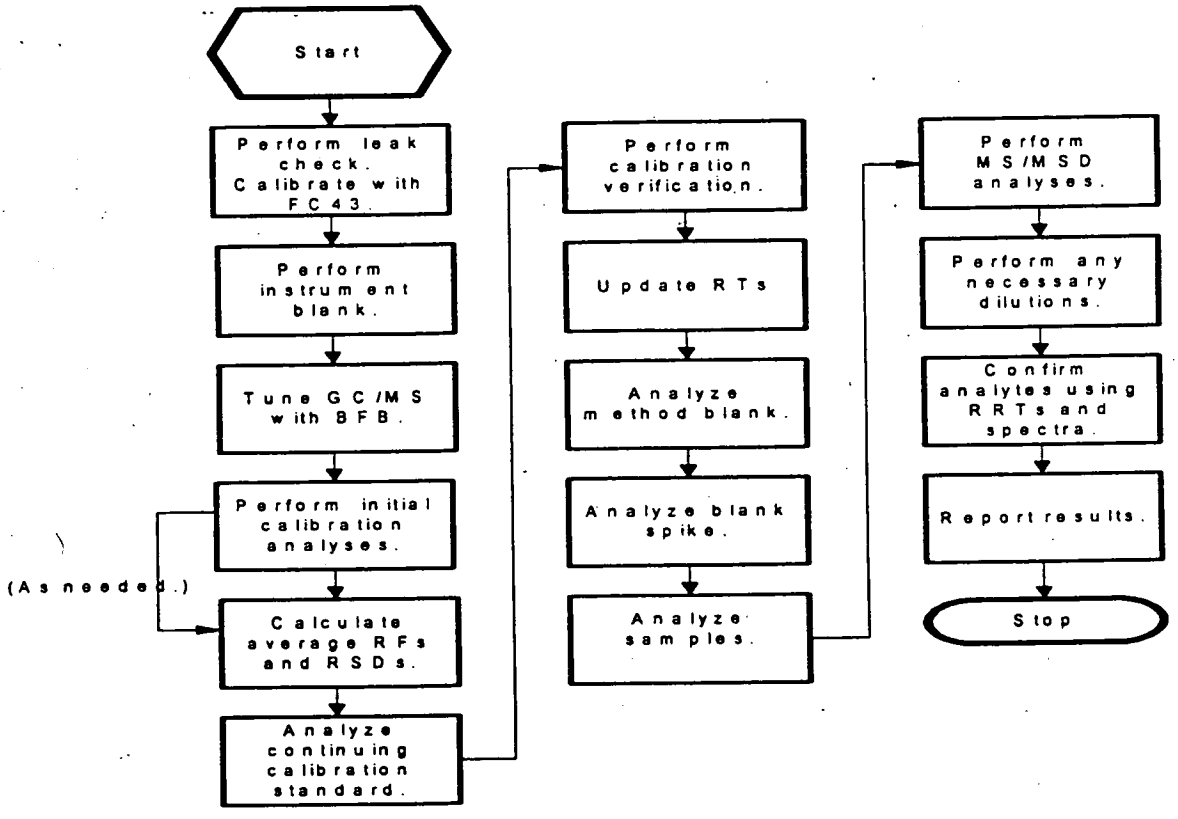
QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Holding Time	14 days from collection.	14 days from collection.	N/A	N/A	HTVR (holding time violation report).
Tuning Verification 5 - 50 ng BFB	Mass Abundance 50 15-40% of 95 75 30-60% of 95 95 100% 96 5-9% of 95 173 < 2% of 174 174 > 50% of 95 175 5-9% of 174 176 95-101% of 174 177 5-9% of 176 *Alternate tuning criteria may be used (e.g.,CLP.524.2).	Mass Abundance 50 8-40% of 95 75 30-66% of 95 95 100% 96 5-9% of 95 173 < 2% of 174 174 50-120% of 95 175 4-9% of 174 176 93-101% of 174 177 5-9% of 176 * Alternate tuning criteria may be used.	Every 12 hours.	Re-tune instrument to meet criteria.	Copy of BFB raw data and tune criteria with file.
Initial Calibration Verification	Minimum of 5 levels with lowest near RL. %RSD for CCCs < 30%. RF for SPCCs: chloromethane 0.10 1,1-dichloroethane 0.10 bromoform 0.10 chlorobenzene 0.30 1,1,2,2-tetrachloroethane 0.30 Non-CCCs: all analytes or average of all analytes = ≤15%.	5 levels 1, 5, 10, 20, 50 µg/L. (Ketones 5,10,20,50,100 µg/L) %RSD for CCCs < 30 %. RFs for SPCCs: chloromethane 0.10 1,1-dichloroethane 0.10 bromoform 0.10 chlorobenzene 0.30 1,1,2,2-tetrachloroethane 0.30 Non-CCCs: all analytes or average of all analytes ≤15%.	Initially.	Reanalysis of out of control standards.	Copies of all raw data, tune file, and Form VI.
Continuing Calibration Verification	Mid-level calibration standard every 12 hours. RF for SPCCs same as initial. RF for CCCs must be < 20% D.	%D for CCC < 20%, Navy work: %D for all other compounds < 40%.	Every 12 hours.	Reanalyze for analytes that exceed the control limits.	Copies of raw data and Form VII.
Internal Standards	Fluorobenzene, chlorobenzene-d5, 1,4-dichlorobenzene-d4, recommended.	Fluorobenzene, chlorobenzene-d5 and 1,4-dichlorobenzene-d4. RT must be ± 30 seconds from last calibration; area must be 50 to 100%.	Every sample.	Reanalyze all samples that do not meet these criteria.	IS areas and RTs are submitted on Form 8, and included in data package.

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Method Blank/ Instrument Blank	One method blank per extraction batch (up to 20 samples) or when there is a change in reagents, whichever is more frequent.	< Reporting limit for all analytes, except for methylene chloride, acetone < 5 X reporting limit.	One method blank per QC period, following the CCV, prior to sample analysis. Typically, an instrument blank is analyzed prior to tuning the instrument.	Samples may not be analyzed until all criteria for the method blank are met.	Copies of raw data filed, and reported electronically. Daily control charts for all method blanks are recorded.
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, and 4-bromofluorobenzene. Percent recoveries must meet established laboratory control limits. See QC database for control limits.	Every sample.	Reanalysis if surrogate recoveries exceed the established control limits.	Surrogates that exceed the control limits are documented in associated file.
Quality Control	One MS/MSD per 20 samples or each batch of samples, whichever is more frequent. Compare % recovery to laboratory established limits.	Blank spike recoveries and RPDs must meet control limits established by laboratory. MS/MSD recoveries are allowed to be out if due to matrix effect. See QC database for control limits.	One MS/MSD per 20 samples. A blank spike is analyzed every QC period.	Reanalysis of MS/MSD unless matrix effect has already been demonstrated. This can be determined by comparing with the blank spike recoveries.	Blank spike and MS/MSD recoveries are stored electronically. Raw data is stored with file. Out of control events are notated in file.
Qualitative Identification	All ions > 10% intensity must be ± 20% of standard; ± 0.06 RRT units of standard RRT.	All ions > 10% intensity must be ± 20% of standard; ± 0.50 min. of standard RT.	For identifying all target analytes in each sample.		Copies of all mass spectra stored with data.

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Sample Preservation and Storage	pH ≤2 with HCL for waters. pH ≤ 2 with sodium bisulfate for low level soils. Medium soils: zero headspace, methanol, or EnCore™ sampler. Store at 4 ° C.	pH ≤2 with HCL for waters. pH ≤2 with sodium bisulfate for low level soils. Medium soils: zero headspace, methanol, or EnCore™ sampler. Store at 4° C.	All water and low level soil samples.	If the water and low level soil samples are not acid-preserved, this is narrated in final report.	pH of all water samples and low level soils, where not preserved to a pH of ≤2, noted in instrument logbook.
Standard Solution Expiration	Stock standards (except gases): 6 months. Store between -10 to -20° C in teflon-sealed liners and amber bottles with minimal headspace, protect from light. Stock gas standards: 2 months. Storage conditions same as stock standards. Calibration standards: daily.	Stock standards (except gases): 6 months. Gas stock standards: 2 months. Stored at -10° to -20° C . Calibration standards: daily. All standards sealed in flame-sealed ampules: 2 years (not to exceed parent expiration date).			All standard data recorded in standard logbook.
Field Sample Amount	2 40 mL vials with zero headspace. Soils: vials, with zero headspace, or a sample aliquot in 40 mL vial. Glass container, septum-sealed screw-caps.	Waters:4 40 mL vials with zero headspace. Soils: 2 oz vials, with zero headspace, or a sample aliquot in 4 -40 mL vial. Glass container, septum-sealed screw-caps.			Bottles received by laboratory documented electronically.
Applicability	Groundwaters, soils, sediments, sludges, non-water miscible wastes and others.				

APPENDIX VI

Method 8260B Flow Chart



Refer to corrective 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

APPENDIX VIII

Method 8260 Optional Analytes for Low Level (25 mL) Water Analyses

Isopropylbenzene	Bromobenzene
1,2,3-Trichloropropane	2-Chlorotoluene
n-Propylbenzene	1,3,5-Trimethylbenzene
4-Chlorotoluene	1,2,4-Trimethylbenzene
tert-Butylbenzene	1,3-Dichlorobenzene
sec-Butylbenzene	p-Isopropyltoluene
1,4-Dichlorobenzene	n-Butylbenzene

Method 8260 Optional Analytes for Low Level (25 mL) Water Analyses

1,2-Dichlorobenzene	1,2,4-Trichlorobenzene
1,2-Dibromo-3-chloropropane	Hexachlorobutadiene
Naphthalene	1,2,3-Trichlorobenzene
Vinyl acetate	

Appendix IX

Additional Appendix IX Compounds (25 mL)

Acrolein	Bromoethane
Allyl chloride	Acrylonitrile
Chloroprene	Methacrylonitrile
Methyl methacrylate	trans-1,4-Dichloro-2-butene
2-Chloroethylvinyl ether	Ethyl methacrylate
Iodomethane	

APPENDIX X

Target Analytes And Their Associated Internal Standards

Fluorobenzene	**Chlorobenzene-d5**	**1,4-Dichlorobenzene-d4**
Dichlorodifluoromethane	Toluene	Isopropylbenzene
Chloromethane	trans-1,3-Dichloropropene	Bromobenzene
Vinyl Chloride	1,1,2-Trichloroethane	1,2,3-Trichloropropane
Bromomethane	Tetrachloroethene	1,1,2,2-Tetrachloroethane
Chloroethane	1,3-Dichloropropane	n-Propylbenzene
Trichlorofluoromethane	2-Hexanone	2-Chlorotoluene
1,1-Dichloroethene	Chlorodibromomethane	4-Chlorotoluene
Acetone	1,2-Dibromoethane	1,3,5-Trimethylbenzene
Carbon disulfide	Chlorobenzene	tert-Butylbenzene
Methylene chloride	1,1,1,2-Tetrachloroethane	1,2,4-Trimethylbenzene
trans-1,2-Dichloroethene	Ethylbenzene	sec-Butylbenzene
1,1-Dichloroethane	m,p-Xylene	1,3-Dichlorobenzene
Vinyl acetate	o-Xylene	1,4-Dichlorobenzene

Fluorobenzene	**Chlorobenzene-d5**	**1,4-Dichlorobenzene-d4**
2,2-Dichloropropane	Styrene	p-Isopropyltoluene
cis-1,2-Dichloroethene	Bromoform	1,2-Dichlorobenzene
2-Butanone		n-Butylbenzene
Bromochloromethane		1,2-Dibromo-3-chloropropane
Chloroform		1,2,4-Trichlorobenzene
1,1,1-Trichloroethane		Naphthalene
Carbon tetrachloride		Hexachlorobutadiene
1,1-Dichloropropene		1,2,3-Trichlorobenzene
Benzene		
1,2-Dichloroethane		
Trichloroethene		
1,2-Dichloropropane		
Dibromomethane		
Bromodichloromethane		
cis-1,3-Dichloropropene		
4-Methyl-2-pentanone		

Additional Appendix IX Compounds And Associated Internal Standard

Fluorobenzene (IS)	**Chlorobenzene-d5**
Acrolein	Ethyl methacrylate
Bromoethane	
Allyl chloride	
Acrylonitrile	
Chloroprene	
Methacrylonitrile	
Methyl methacrylate	
trans-1,4-Dichloro-2-butene	
Iodomethane	
2-Chloroethylvinyl ether	

APPENDIX XI

Target Analytes And Their Quantitation Ions

Compound	Primary Quant Ions*	Secondary Quant Ions*
Dichlorodifluoromethane	85	87
Chloromethane	50	52
Vinyl Chloride	62	64
Bromomethane	96	94
Chloroethane	64	66
Trichlorofluoromethane	101	103
1,1-Dichloroethene	96	61,63
Acetone	43	58
Carbon disulfide	76	78
Methylene chloride	84	86
trans-1,2-Dichloroethene	96	98,61
1,1-Dichloroethane	63	65,83
Vinyl acetate	43	86
2,2-Dichloropropane	77	97
cis-1,2-Dichloroethene	96	98,61
2-Butanone	43	57
Bromochloromethane	128	49,130
Chloroform	83	85
1,1,1-Trichloroethane	97	99,61
Carbon tetrachloride	117	119
1,1-Dichloropropene	75	77,110
Benzene	78	-
1,2-Dichloroethane	62	98
Trichloroethene	95	97,130,132
1,2-Dichloropropane	63	112
Dibromomethane	93	95,174
Bromodichloromethane	83	85,127
cis-1,3-Dichloropropene	75	77
4-Methyl-2-pentanone	43	58,85
Toluene	92	91

Compound	Primary Quant Ions*	Secondary Quant Ions*
trans-1,3-Dichloropropene	75	77
1,1,2-Trichloroethane	83	97,85
Tetrachloroethene	166	164
1,3-Dichloropropane	76	78
2-Hexanone	43	58,100
Chlorodibromomethane	129	208,206
1,2-Dibromoethane	107	109
Chlorobenzene	112	114
1,1,1,2-Tetrachloroethane	131	133
Ethylbenzene	91	106
m,p-Xylene	106	91
o-Xylene	106	91
Styrene	104	78
Bromoform	173	175
Isopropylbenzene	105	120
Bromobenzene	156	158
1,2,3-Trichloropropane	75	77
1,1,2,2-Tetrachloroethane	83	85
n-Propylbenzene	120	91
2-Chlorotoluene	126	91
4-Chlorotoluene	91	126
1,3,5-Trimethylbenzene	105	120
tert-Butylbenzene	91	134,119
1,2,4-Trimethylbenzene	105	120
sec-Butylbenzene	105	134

Compound	Primary Quant Ions*	Secondary Quant Ions*
1,3-Dichlorobenzene	146	148,111
1,4-Dichlorobenzene	146	148,111
p-Isopropyltoluene	119	134,91
1,2-Dichlorobenzene	146	148,111
n-Butylbenzene	91	92
1,2-Dibromo-3-chloropropane	75	155,157
1,2,4-Trichlorobenzene	180	182
Naphthalene	128	-
Hexachlorobutadiene	225	223,227
1,2,3-Trichlorobenzene	180	182

Additional Appendix IX Compounds And Their Quantitation Ions

Compound	Primary Quant Ions*	Secondary Quant Ions*
Acrolein	56	55,58
Bromoethane	108	-
Allyl chloride	41	78
Acrylonitrile	53	52,51
Chloroprene	53	51
Methacrylonitrile	41	39
Ethyl methacrylate	69	-
Methyl methacrylate	41	39
trans-1,4-Dichloro-2-butene	53	75
Vinyl acetate	43	86
Iodomethane	142	127
2-Chloroethylvinyl ether	63	65

* Primary and secondary quantitation ions assigned by Laucks may be slightly different than those recommended in SW 846, in order to avoid interference.

APPENDIX XII

Target Analytes, Their Elution Order And Standard Mixes

Compound	Standard Mix
Dichlorodifluoromethane	6
Chloromethane	6
Vinyl Chloride	6
Bromomethane	6
Chloroethane	6
Trichlorofluoromethane	6
Acrolein	8
1,1-Dichloroethene	5
Acetone	7
Iodomethane	9
Bromoethane	10
Carbon disulfide	11
Allyl Chloride	12
Methylene chloride	5
trans-1,2-Dichloroethene	5
Acrylonitrile	13
1,1-Dichloroethane	4
Vinyl acetate	14
Chloroprene	15
2,2-Dichloropropane	4
cis-1,2-Dichloroethene	5
2-Butanone	7
Methacrylonitrile	12
Bromochloromethane	4
Chloroform	4
1,1,1-Trichloroethane	4
Carbon tetrachloride	4
1,1-Dichloropropene	3
1,2-Dichloroethane-d4 (surr)	18
Benzene	2
1,2-Dichloroethane	3
Trichloroethene	3

Compound	Standard Mix
1,2-Dichloropropane	3
Dibromomethane	4
Methyl methacrylate	12
Bromodichloromethane	5
2-Chloroethylvinyl ether	16
cis-1,3-Dichloropropene	3
Ethyl methacrylate	12
4-Methyl-2-pentanone	7
Toluene-d8 (surr)	18
Toluene	2
trans-1,3-Dichloropropene	3
1,1,2-Trichloroethane	3
Tetrachloroethene	4
1,3-Dichloropropane	3
2-Hexanone	7
Chlorodibromomethane	5
1,2-Dibromoethane	3
Chlorobenzene	1
1,1,1,2-Tetrachloroethane	3
Ethylbenzene	2
m,p-Xylene	1,2
o-Xylene	1
Styrene	2
Bromoform	4
Isopropylbenzene	1
4-Bromofluorobenzene (surr)	18
Bromobenzene	2
1,2,3-Trichloropropane	3
1,1,2,2-Tetrachloroethane	3
n-Propylbenzene	1
trans-1,4-Dichloro-2-butene	17
2-Chlorotoluene	1
4-Chlorotoluene	1
1,3,5-Trimethylbenzene	2
tert-Butylbenzene	1
1,2,4-Trimethylbenzene	2

Compound	Standard Mix
sec-Butylbenzene	1
1,3-Dichlorobenzene	1
1,4-Dichlorobenzene	1
p-Isopropyltoluene	2
1,2-Dichlorobenzene	1
n-Butylbenzene	2
1,2-Dibromo-3-chloropropane	3
1,2,4-Trichlorobenzene	2
Naphthalene	2
Hexachlorobutadiene	3
1,2,3-Trichlorobenzene	2

Key:

1 = Supelco VOC Mix 1
2 = Supelco VOC Mix 2
3 = Supelco VOC Mix 3
4 = Supelco VOC Mix 4
5 = Supelco VOC Mix 5
6 = Supelco VOC Mix 6
7 = Supelco TCL Mix 1
8 = From neat
9 = Supelco Single std
10 = Chem Service Single Standard
11 = Supelco single standard
12 = Supelco 8240B Mix 7
13 = Supelco single std
14 = Supelco single standard
15 = Neat Material
16 = Supelco Single standard
17 = Supelco Single standard
18 = Neat material

At the time of this writing, these are the current standard solutions used for the calibration standards. However, these standards may change due to Vendor supply or project-specific requirements.

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-8302

Title: HPLC Ordnance Data Review

Revision history:

Number	Date
0	07/31/95
1	01/23/98

Revised by: Monica Carr, Organic Division Manager Date: 1/30/98

Approved by: Harry Romberg, QC Officer Date: 1-30-98

Approved by: Karen Kotz, Laboratory Director Date: 1/26/98

UNCONTROLLED

Summary

The data reviewer will be working on reviewing data packages which have been completed by the HPLC analyst. The review will follow this procedure, utilizing reference materials attached to this procedure as well as client/project specific requirements (such as Maximum % RSD and CRDLs, if different from EPA PQLs), which will be provided by the HPLC Supervisor. All data review will be documented on the "Data Review Checklist" (attached to this procedure) and comments will be referenced to the Review Item and number. At the end of the review process, the reviewer will arrange the data package in the order specified here, with the completed "Data Review Checklist" and accompanying notes from the analyst(s) (if applicable) on the top of the package. This package will be given to the HPLC Supervisor for final review and narrative preparation.

List of Attachments

1. Laucks Testing Labs - Data Review Checklist [DRC] (HPLC - Ordnance Version)

Procedure

1. Initial Information

   A. Before beginning the review process, it is important to confirm with the HPLC Supervisor what method was used and what the associated QC limits are. Most of this information is printed on the following forms, however it must be confirmed that these QC limits are correct for the method/client/project. The levels for the following QC criteria should be confirmed:

Initial Calibrations:	Maximum %RSD	*(usually 20%, will be on Form V)*
Continuing Cals:	Maximum %D	*(usually 15%, will be on Form VII & ICV)*
Surrogates:	Acceptable Recovery Range *(will be on Form II)* and spiking levels *[100 ul of Surrogate Spike Soln. Residue levels for Water and Soil will be 2 ppm.]*	
Blank:	Maximum Allowable Concentration for confirmed compounds	
MS/MSD:	QC Limits for % Rec and % RPD *(on Form III)* and spiking levels *[100 ul of Spiking Solution. Water will be 4 ppb, Soil will be 133 ppb.]*	

   B.

Laucks forms in the data package	Laucks #	*EPA Equivalent*
QC Summary Data:		
"Surrogate Recovery Summary Report"	SURR GC	*Form II*
"MS/MSD Report" *and Blank Spikes/Duplicates*	BLKSPK-1	*Form III*
	BLKSPK-2	
	MS/MSD-1	
	MS/MSD-2	
"Method Blank Summary"	BLK SUMM	*Form IV*
Sample Data: *(and Raw QC Data)*		
"Organics Analysis Data Sheet"		*Form I*
"Compound Confirmation"	GC CONF	*Form X*

B. Laucks forms in the data package *(continued)*

	Laucks #	*EPA Equivalent*
Standards Data:		
"Initial Multi-point Calibration"	ICAL GC	*Form VI*
Cal. Factors:	Part 1	
RTs:	Part 2	
Peak Response (Areas):	Part 3	
Linear Regression:	Part 4	
"Initial Calibration Verification Worksheet"	ICV-1	
"Continuing Calibration Verification Worksheet"	CCV-1	*Form VII*
Calibration Factor:	Page 1	
Retention Times:	Page 2	

2. Overall Review

   A. Review Narrative already written by analyst to familiarize yourself with the project and to confirm what comments have already been written.

<u>QC Summary</u>

3. Form II ("Surrogate Recovery Summary Report")

   A. Confirm all sample and QC are present by comparing to the bench sheet. *Check "Yes" or "No" on DRC #A1. Discrepancies must be noted on the DRC, referencing #A1.*

   B. Check to make sure the recoveries for surrogates are within QC limits. Outliers will be marked with an "*". Comment on any that are outside limits or are not present (have been diluted out). *Check "Yes" or "No" on DRC #A2 at completion of review. If any recoveries are outside QC Limits, these must be noted on the DRC, referencing #A2.*

4. Form III ("MS/MSD Report" *and Blank Spikes/Duplicates* BLKSPK-1,2, MS/MSD-1,2)

   A. **BLKSPK-1 and/or MS/MSD-1** - Confirm the spike added against the normal levels of 4 ppb for water, 133 ppb for soil. If not the normal levels, confirm against the bench sheets and log books.

   B. **BLKSPK-1 and/or MS/MSD-1** - Check the amount found against Form I's (these are entered manually on Form III's) for the corresponding sample (check Lab ID against bench sheet). Check to see if any % Rec, and/or RPD's are out of control (will be indicated with an "*"). *Check "Yes" or "No" on DRC #A3 and #A4 at completion of review. The number and type (high or low) of out of controls must be noted on the DRC, referencing #A3 and/or #A4 followed by the Lab ID # where the out of controls occurred.*

   C. **BLKSPK-2 and/or MS/MSD-2** - Check that the correct samples are associated with the blank spike and/or MS/MSD by confirming against the bench sheet. *Check "Yes" or "No" on DRC #A5 at completion of review. Discrepancies must be noted on the DRC, referencing #A5.*

5. Form IV ("Method Blank Summary")

   A. Check the blank name and corresponding lab ID numbers against the bench sheet.
   Check of "Yes" or "No" on DRC #A6. Discrepancies must be noted on the DRC, referencing #A6.

Standards Data

7. Form VI ("Initial Multi-point Calibration" ICAL HPLC

Cal. Factors:	Part 1
RTs:	Part 4
Peak Response (Areas):	Part 2
Linear Regression:	Part 3
Amount Summary:	Part 5

   A. **Part 1** - Check that % RSD is within 20% limits (will be marked with a "*" if outside limit). Also confirm that the lab file ID's match those on the Target Sequence for the ICAL.
   Check "Yes" or "No" on DRC #C2 at completion of review. Discrepancies must be noted on the DRC, referencing #C2.

8. "Initial Calibration Verification Worksheet" ICV-1

   A. There may be an ICV Form for the mid-point standard from the ICAL, but only if samples were analyzed directly after the ICAL, before another CCV. Check that there is an ICV associated with each analytical batch of samples reported here by checking date and time against Form IV ("Method Blank Summary"). If unclear as to which ICVs correspond to the samples, the Target Sequence should be referenced.
   Check "Yes" or "No" on DRC #C1 at completion of review. Discrepancies must be noted on the DRC, referencing #C1.

   B. Confirm that %D's are within limits (15%). They will be marked with a "*" if out.
   Discrepancies must be noted on the DRC, referencing #C1.

9. Form VII ("Continuing Calibration Verification Worksheet" CCV-1)

Calibration Factor:	Page 1
Retention Times:	Page 2

   A. **Page 1** - Check that % D is within 15% control limits. Confirm that all forms are included here by comparing against the Target Sequence.

   B. **Page 2** - Confirm Continuing Calibration RTs are within RT window (note: these will **not** be marked with a "*" if out of control).

   Check "Yes" or "No" on DRC #C4 at completion CCV of review. Discrepancies must be noted on the DRC, referencing #C4.

10. Chromatograms and Processed Files
   A. Confirm that chromatograms and processed files for all calibration standards and CCVs are present.
   Check "Yes" or "No" on DRC #C5 at the completion of review. Discrepancies must be noted on the DRC, referencing #C5.

Sample Data and Raw QC Data

12. Form I ("Organics Analysis Data Sheet")

A. Check dates received and dates of collection against the SDG Database Report, and date extracted against the extraction bench sheets. Confirm that the dates extracted and analyzed were within holding times.
Extraction: 14 days for soils and 7 days for waters (measured from date of collection).
Analysis: 40 days from extraction.
Check "Yes" or "No" on DRC #B1 at the completion of review. Discrepancies must be noted on the DRC, referencing #B1.

B. Check sample size against the bench sheet - the initial sample (there are sometimes many dilutions) will always be a 1:2 dilution for water and soil. Confirm that the percent moisture is correct by comparing to the SDG report (for soils). Waters should always be 100% moisture.
Check "Yes" or "No" on DRC #B2 at the completion of review. Discrepancies must be noted on the DRC, referencing #B2.

C. Check that all "hits" are correctly transferred to Form I from Form X (unless they are crossed out on Form X). In addition, check the retention times of the analyte to ensure that they are within the RT window. The retention times and RT windows are located on the compound confirmation sheet.
Check "Yes" or "No" on DRC #B3 at the completion of review. Discrepancies must be noted on the DRC, referencing #B3.

D. Were all samples with analyte concentrations > the highest calibration standard diluted and reanalyzed? If so, were the diluted sample results linear with the last result that exceeded calibration (i.e. within 25%)?
Check "Yes", "No" or "N/A" on DRC #B4 at completion of review. Discrepancies must be noted on the DRC, referencing #B4.

E. Check that the sample number on Form I matches that on the chromatogram.
Check "Yes" or "No" on DRC #B5 at the completion of review. Discrepancies must be noted on the DRC, referencing #B5.

F. Check that flags are used correctly. Flags not referenced on the "Use of Data Qualifiers" Memo are detailed below.
"D" These flags are used on diluted sample results, but are not used on the initial 1:2 dilution as this is a dilution of methanol with water to produce the appropriate sample matrix for HPLC analysis.
"E" Anything above 1000 ppb extract concentration on Form X must be flagged with an "E" on Form I and re-diluted to attain an accurate analysis within the calibration curve.
"X" This will be used for the most accurate "hit" from all the dilutions. Only one "X" for each sample/analyte should occur throughout the dilutions, as there is only one "most accurate" hit.
"Z" A "Z" flag on sample report forms indicates coelution has occurred between two or more target analytes on the confirmation CN column. For this reason, quantitative confirmation is not possible.
Discrepancies must be noted on the DRC, referencing #B6.

13. Form X ("Compound Confirmation")

    A. Check that any %D over 25% is "P" flagged on Form I (note that blank and matrix spikes are not confirmed and %D's reported should be crossed out for blank spikes).
    Check "Yes" or "No" on DRC #B6 at the completion of review of data qualifiers. Discrepancies must be noted on the DRC, referencing #B6.

    B Check the retention time windows located on the the compound confirmation form, with the copy of the retention time window study located in the Bench Sheet section of the data package.
    Check "Yes" or "No" on DRC #B7 at the completion of review of data qualifiers. Discrepancies must be noted on the DRC, referencing #B7.

At the end of the Sample Data and Raw QC review, remove all Form Xs and place them at the end of the Standards Data set.

14. Final Review
    A. Were recoveries for the SRM (if required by client/project) within QC Limits?
    Check "Yes", "No" or "N/A" on DRC #D2 at completion of review. Discrepancies must be noted on the DRC, referencing #D2.

    B. Are all nonconformances included and noted on the DRC?
    Check "Yes" or "No" on DRC #D1 at completion of review. Discrepancies must be noted on the DRC, referencing #D1.

17. Order of Final Packet - All in numerical and chronological order and in order by column
    Check "Yes" or "No" on DRC #D3 at review completion. Note discrepancies referencing #D3.

    1. QC Summary
       Forms II through IV

    2. Sample Data
       For each sample, the following packet:
       All Form I's, then chromatograms and integration reports *(C18 first, then CN column)*

    3. Standard Data
       Form VI's (Parts 1,2,3, and 4) *(C18 column first, then CN column on each)*
       ICV-1 (Initial Calibration Verification Sheets)
       Form VII's (Pages 1 and 2) *(C18 column first, then CN column on each)*
       Form X's *(samples first, followed by QC samples)*
       Raw Standards Data, *(compound confirmation, chromatograms and integration reports)* including:
       Multi-point calibrations *(C18 column, then CN column)*
       All CCVs and ICVs in chronological order
       MDL Standard
       I Blanks

4. Raw QC Data *(Method Blanks, Blank Spikes/Blank Spike Duplcts, MS/MSDs, Control charts.)*
   For each sample, the following packet:
   All Form I's, then chromatograms and integration reports *(C18 first, then CN column)*

5. Bench Sheets
   Including: SDG Report, Copy of instrument logbook, Extraction bench sheets, Logbooks for surrogates/spikes (referenced on bench sheet), Logbooks for standards, RT Study.

Attachment 1
Data Review Checklist

Laucks Testing Labs Data Review Checklist
Ordnance 8330 - HPLC Department

Work Order Number:	
Analysis Batch Dates:	
Method:	SDG #:

Review Item	Yes	No	N/A	2nd Review
A. QC Summary 1. Are all required QC results present?				
2. Do surrogate recoveries meet QC limits?				
3. Are blank spike/blank spike duplicate results correct and do they meet QC %Rec and RPD limits?				
4. Are MS/MSD results correct and do they meet QC %Recovery & RPD limits?				
5. Are correct samples associated with the blank spike(s) and/or MS/MSDs?				
6. Are all blanks reported?				
7. Is MDL recovery within QC limits?				
B. Sample Data and Raw QC Data 1. Were samples extracted and analyzed within holding times?				
2. Are sample sizes and % moisture correct?				
3. Are all hits recorded correctly?				
4. Were samples diluted as required and were dilutions linear (within 25% of last value)?				
5. Were sample ID's checked?				
6. Were data qualifier flags used correctly?				
7. Were the retention time windows on the compound confirmation form recorded properly? Review RT study located in Bench Sheet section.				
C. Standards Data 1. Is there an ICV run every day and are all %Ds within QC limits?				
2. Are calibration %RSDs within QC limits?				
3. Are all ICVs and CCVs recorded and are RT %Ds within QC control limits?				
4. Are CCV %Ds and RTs within QC limits?				
5. Is all raw data present?				
D. Final Review 1. Are all nonconformances listed?				
2. Are recoveries for the SRM within limits?				
3. Are all components of the data package present and in correct order?				

Laucks Testing Labs Data Review Checklist
Ordnance 8330 - HPLC Department

Work Order Number:	
Analysis Batch Dates:	
Method:	SDG #:

Comments on any "No" responses:

_____
_____
_____
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Data Reviewer: _____ **Date:** _____

2nd Level Reviewer: _____ **Date:** _____

LAUCKS TESTING LABORATORIES INC.

Seattle, Washington

SOP #:LTL-8330

Determination of Nitroaromatics and Nitramines by SW-846 Method 8330

Revision history:

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UNCONTROLLED

Revised by: Monica Carr, Organic Division Manager — Date: 1/25/99

Approved by: Harry Romberg, QA Officer — Date: 1-25-99

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1. Introduction and Scope

1.1 Method Description

1.1.1 This method is used for the trace analysis of ordnance compounds in water, soil, and sediment samples by high performance liquid chromatography. The concentrated water sample extracts are diluted 1:1 (v/v) with reagent grade water prior to analysis. The sample extracts are analyzed using a C18 (octadecyl) reverse phase column, and target analyte concentrations are measured at either 254 nm or 210 nm using a UV detector. All positive measurements observed on the C18 column are confirmed by a second analysis which uses a CN (cyano) column. The C18 column is considered the primary column and is used for quantitation of all target analytes.

1.1.2 This method is used to determine part per billion levels of the ordnance analytes listed below:

Compound	Acronym
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine	HMX
Hexahydro-1,3,5-trinitro-1,3,5-triazine	RDX
1,3,5-Trinitrobenzene	1,3,5-TNB
1,3-Dinitrobenzene	1,3-DNB
Methyl-2,4,6-trinitrophenylnitramine	Tetryl
Nitrobenzene	NB
2,4,6-Trinitrotoluene	2,4,6-TNT
4-Amino-2,6-dinitrotoluene	4-Am-DNT
2-Amino-4,6-dinitrotoluene	2-Am-DNT
2,4-Dinitrotoluene	2,4-DNT
2,6-Dinitrotoluene	2,6-DNT
2-Nitrotoluene	2-NT
3-Nitrotoluene	3-NT
4-Nitrotoluene	4-NT

6 Additional Compounds
(Separate Analysis at 254 nm)

Compound	Acronym
2,4-Diamino-6-nitrotoluene	2,4-DA-6-NT
2,6-Diamino-4-nitrotoluene	2,6-DA-4-NT
3,5-Dinitroaniline	3,5-DNA
1-Nitroso-3,5-dinitro-1,3,5-hexahydrotriazine	MNX
1,3,5-Trinitroso-1,3,5-hexahydrotriazine	TNX
2,2',6,6'-Tetranitro-4,4'-azoxytoluene	4,4'-TN-AZOXY

PETN/NG Compounds
(Separate Analysis at 210 nm)

Compound	Acronym
Nitroglycerin	NG
Pentaerythritoltetranitrate	PETN

1.1.3 Aqueous samples of higher concentration can be directly analyzed by diluting 1:1 (v/v) with methanol or acetonitrile, filtering, separating on a C18 reverse phase column, and determined at either 254 nm or 210 nm, and confirmed on a CN column.

1.1.4 This method is restricted to use by, or under the supervision of, analysts experienced in the use of high pressure liquid chromatography and in the interpretation of chromatograms. Each analyst performing this method must have demonstrated the ability to perform the described chromatographic analysis and/or data interpretation.

1.2 Method Deviations & Comments

1.2.1 The following items represent deviations from the published version of method SW 8330 which are followed as standard operating procedure in the performance of this method at Laucks.

1. Single injections of calibration standards are analyzed rather than triplicate injections in a random order. It has been determined at Laucks that single injections of these standards yield acceptable calibration and linearity data as evidenced by the calculated percent RSDs used to evaluate the initial calibration data.

2. Method SW 8330 specifies that all analyses are to be performed using a mobile phase which consists of a 50:50 mixture of methanol/water under

isocratic conditions. Laucks employs a gradient elution program (detailed in Section IV) in order to improve the separation of the target analytes on the CN column.

3. According to Method 8330, all working standards are to be prepared daily in a methanol/calcium chloride solution. The practice at Laucks for 8330 and the 6 additional compounds is to prepare working standards using acetonitrile and diluting 50/50 with water just prior to analysis. NG/PETN are prepared in a 50/50 mixture of acetonitrile and water. These working solutions have been demonstrated to be stable for at least 6 months. The stability of target analyte responses in the working solutions in use (especially that of tetryl) is used to determine whether new solutions should be prepared.

4. Tetryl decomposes rapidly in methanol/water solutions, as well as with heat. Laucks has experienced erratic recovery of tetryl from spiked sample extracts.

5. The confirmation column demonstrates full or partial coelution for the following target analyte pairs: 1,3-dinitrobenzene and 1,3,5-trinitrobenzene; 2-nitrotoluene, 3-nitrotoluene and 4-nitrotoluene; and 2,4-dinitrotoluene, 2,6-dinitrotoluene and 2,4,6-trinitrotoluene. Therefore, positive confirmation can not be made when two or more co-eluting peaks have been tentatively identified on the primary column.

6. At the time of this writing, analysis of the 6 additional compounds (not including PETN/NG) combined with the 14 ordnance compounds results in the co-elution of 2,6-DA-4-NT and HMX; and 3,5-DNA and tetryl on the C18 column using the 8330 method. However, tetryl and HMX do not co-elute with any attenuation compound on the CN column using the 8330 method. These two compounds (HMX and tetry) do not co-elute with any of the attenuation compounds on the primary colum nor confirmation column using the attenuation method.

7. Although the compounds NG and PETN are not listed in SW 846 Method 8330, Laucks has found that adequate recovery of these compounds can be achieved using this method by modifying the wavelength from 210 nm to 254 nm.

8. Picric and picramic acids exhibit substantial peak shift and may elute near or co-elute with HMX on the C18 column using the 8330 method. However, no attenuation compounds, nor NG/PETN coleute on the primary (PAH)

column using the picric/picramic method. The buffer is used in this method in order to stablize these compounds.

1.2.2 In order to attain lab-wide consistency among staff members for decision-making processes with regard to laboratory anomalies, several common items have been addressed in this SOP. Any occurrences which are not covered in this SOP should be discussed with the supervisor, prior to implementing a solution.

1.2.3 One example is the determination of potential carry-over in sample analyses. Any samples analyzed subsequent to a high level sample (which is defined by yielding one or more target analytes above the calibration range) should be thoroughly examined for potential carry-over of the same target analyte(s). Corrective action in the form of reanalysis for possible carry-over should be performed and documented in the narrative.

1.2.4 Another example is the review of all sample chromatograms for analytes which may not show up on the sample quantitation report due to data system error or retention time shift. All peaks should be examined and evaluated based on the retention times and sample concentration in order to prevent reporting false negatives.

1.3 Sample Collection, Sample Storage, Holding Times

1.3.1 Samples are collected in amber glass containers with Teflon-lined caps. All samples and sample extracts are stored at 4°C ±2°C. According to guidelines listed in SW-846 for the extraction of semivolatile compounds, water samples must be extracted within 7 days of collection, and soil samples must be extracted within 14 days of collection. All extracts must be analyzed within 40 days of sample preparation.

1.3.2 Although not a routine sampling practice, a soil holding time study performed by the U.S. Army Cold Regions Research and Engineering Laboratory recommends a maximum holding time of eight weeks when samples are frozen.

1.3.3 A similar study performed for water samples recommends a maximum holding time of 50 days for relatively sterile samples which are refrigerated. Surface waters, or samples likely to have significant microbial activity, may suffer significant losses of nitroaromatic compounds (particularly trinitrobenzene and trinitrotoluene) within 1-2 days, even under refrigeration. If microbial activity is suspected, water samples should be extracted as soon after collection as is practical.

1.4 Definition of Terms

1.4.1 This section defines terms and acronyms as they are used in this SOP. Other terms, such as MS/MSD or method blank, are not defined here since it is assumed that the user of this SOP already understands their more general meaning.

Method Blank Spike	A background free matrix (DIW for water, clean sand for soils/sediments) to which known amounts of target analytes and surrogates are added each time sample extracts are prepared. In the context of this SOP, a blank spike is the same as a QC check standard. See also QC check standard.
CCV	Continuing calibration verification. This is the same acronym used in the CLP program. This is a standard injected at some prescribed frequency during the analysis sequence to determine whether the instrument has remained in calibration.
CF	Calibration factor. The ratio of peak response to nanograms injected. This term is defined in the same way in both the CLP contract and SW-846.
DIW	Deionized water. Lab reagent water. Organic-free water. Since the systems used to provide DIW at Laucks all contain carbon polishing filters, they are capable of providing organic-free water for use in method blanks and method blank spikes.
IBLK	An instrument blank is solvent containing the method surrogates and is injected into the instrument to monitor for carryover between sample extract injections.
ICV	Initial calibration verification. It is a standard which is injected at the start of each QC period that is compared to the initial multi-point calibration to determine whether the instrument is still in calibration.
IDL	Instrument detection limit. The lowest concentration of a target analyte that will yield a signal:noise ratio of at least 3x. Used as a starting point for selecting MDL study spiking levels.

MDL	Method detection limit. The lowest concentration in a sample which will yield a positive result that is greater than zero at a known level of confidence. MDLs are empirically determined at Laucks.
QC Check Standard	Quality control check standard. Referred to in this SOP as a blank spike. A QC check standard is a requirement of SW-846 method 8000 and is used to determine whether the analytical system is in control if MS/MSD recoveries are out of control. See also blank spike.
QC Period	Quality control period. An analysis sequence initiated by the injection of one or more standards, followed by sample extracts, and terminated with a standard injection. A QC period can be open-ended chronologically, but calibration verification must be documented using the procedures in this SOP.
RSD or %RSD	Relative standard deviation or percent relative standard deviation. The ratio of the standard deviation of a set of values to the mean of the set of values. A measure of the similarity of the values one to another.
RT	Retention time. The time (in minutes) at which a target analyte elutes from the LC column.
RT Window	Retention time window. The ± value which is applied to the ICV to establish the time range used to make tentative compound identifications.
Sequence	A set of sample extracts and standard solutions injected into an instrument in a chronologically continuous group. See also QC period.

2. Equipment List and Standards

2.1 Chromatographic System

Primary Column:	C18 (octadecyl) reverse phase HPLC column, 15 cm x 4.6 mm, 5 µm particle size, (Rainin Microsorb or equivalent).

Secondary Column:	CN (cyano) reverse phase HPLC column, 25 cm x 4.6 mm, 5 µm particle size, (Supelcosil LC-CN or equivalent). C8 and CN reverse phase HPLC columns in series, 10cm x 3.9mm, 4µm particle size, (Waters Nova- Pak or equivalent). These are used in the for the analysis of the 6 additional compounds.
Mobile Phase:	Methanol (EM Science brand high purity solvent or equivalent). Reagent water (Modulab Polisher HPLC grade water or equivalent).
UV Detector:	254 nm (210 nm for NG and PETN)
HPLC System:	Rainin HPLC system - HPXL solvent delivery system capable of achieving 4000 psi. 50 µL sample loop. Knauer variable wavelength UV detector. Dynamax automatic sample injector. Digital integrator: EZChrom.
Waters System:	Waters 712 WISP Sample Processor or equivalent. 50 µL sample loop or equivalent. Waters 486 tunable absorbance detector or equivalent. Waters 600E Multisolvent delivery system or equivalent. Digital Integrator: EZChrom.
Hewlett Packard System:	HP 1313A Autosampler. HP G1311A QuatPump. HP G1322A On-line degasser. HP G1316A Thermostat column compartment. HP 1314A Variable wavelength detector. Digital Integrator: EZChrom.

2.1.1 Column Temperature Control:

2.1.2 Column temperature is controlled through the use of a column heater which is maintained at a temperature of 25°C.

2.2 Standards

2.2.1 Target Analyte Stock Solution

2.2.1.1 The stock solution used contains the 14 standard compounds in the mix at a concentration of 1000 µg/mL and is generally purchased from AccuStandard Inc. (25 Science Park, New Haven CT 06511). Equivalent solutions from this or other vendors are also acceptable.

2.2.1.2 The NG and PETN stock solutions are purchased individually from AccuStandard, Inc. (25 Science Park, New Haven, CT 06511). The NG solution comes in 1 mL ampules at 4000 µg/mL in ethanol. The PETN solution comes in 1 mL ampules at 1000 µg/mL in methanol. Equivalent solutions from this or other vendors are also acceptable.

2.2.1.3 The 3,5-Dinitroaniline and 1,3,5-Trinitroso-1,3,5-hexahydrotriazine standards are made from standard analytical reference materials obtained from the U.S. Army Environmental Center (Aberdeen Proving Ground, MD 21010). The 2,4-Diamino-6-nitrotoluene, 2,6-Diamino-4-nitrotoluene and 2,2',6,6'-Tetranitro-4,4'-azoxytoluene standards are made from standard analytical reference materials obtained from AccuStandard, Inc. (25 Science Park, New Haven, CT 06511). The 1-Nitroso-3,5-dinitro-1,3,5-hexahydrotriazine standards are made from standard analytical reference materials obtained from Stanford Research.

2.2.1.4 Calibration standard, surrogate stock and IBLK surrogate solutions are detailed in Appendix I.

2.2.2 Surrogate Stock Solution

2.2.2.1 The surrogate stock solution is received in a methanolic solution at a concentration of 1000 µg/mL. The surrogate can also be prepared by weighing 100 mg of 1,2-dinitrobenzene into a 100 mL volumetric flask and diluting to volume with methanol to yield a concentration of 1000 µg/mL.

2.2.3 IBLK Working Solution

2.2.3.1 The working instrument blank solution contains the surrogate compound only at a concentration of 2.0 µg/mL. Prepare this solution by adding 100 µL of the 1000 µg/mL surrogate stock solution to a 50 mL volumetric flask. Dilute to volume so that the final solvent concentration is methanol/water at a ratio of 1:1.

2.2.4 Working Calibration Standards

2.2.4.1 Prepare the working calibration standards for the 14 standard analytes in the following manner. All solutions are prepared in acetonitrile and diluted 1:1 with water just prior to analysis. These 14 analytes are combined into one working solution, since they do not co-elute on the primary column.

Standard	Source Solution	Amount Added (mL)	Final Volume (mL)	Final Concentration (µg /mL)
Standard #6	A	0.20	10	20.0
	B	0.20		
Standard #5	A	0.1	10	10.0
	B	0.1		
Standard #4	C	1.0	10	2.0
Standard #3	D	1.0	10	1.0
Standard #2	E	1.0	10	0.2
Standard #1	F	1.0	10	0.1

Source Solution	Concentration (µg /mL)
A = 14 Component Mix	1000
B = Surrogate Stock Solution	1000
C = Standard #6	20
D = Standard #5	10
E = Standard #4	2.0
F = Standard #3	1.0

2.2.4.2 Prepare the working calibration standards for the 6 additional analytes in the following manner. All solutions are prepared in acetonitrile and diluted 1:1 with water just prior to analysis.

Standard	Source Solution	Amount Added (mL)	Final Volume (mL)	Final Concentrations (µg /mL)
Standard #6	A	2.0	10	20.0
	B	0.2		
	C	0.2		
	D	0.2		
	E	0.2		
	F	2.0		
	G	0.2		
Standard #5	H	5.0	10	10.0
Standard #4	H	1.0	10	2.0
Standard #3	I	1.0	10	1.0
Standard #2	J	1.0	10	0.2
Standard #1	K	1.0	10	0.1

Source Solution	Concentration (µg/mL)
A = MNX	100
B = TNX	1000
C = 3,5-DNA	1000
D = 2,6-DA-4-NT	1000
E = 2,4-DA-6-NT	1000
F = 4,4'-TN-AZOXY	100
G = 1,2-DNB	1000
H = Standard 6	20.0
I = Standard 5	10.0
J = Standard 4	2.0
K = Standard 3	1.0

2.2.4.3 Prepare the working calibration standards for PETN/NG in the following manner. All solutions are prepared in a 1:1 acetonitrile/water mixture.

Standard	Source Solution	Amount Added (mL)	Final Volume (mL)	Final Concentrations NG:PETN:1,2-DNB (µg /mL)
Standard #5	A	0.05	10	20.0
	B	0.10	10	10.0
	C	0.10	10	10.0
Standard #4	D	5.0	10	10.0, 5.0, 5.0
Standard #3	E	5.0	10	5.0, 2.5, 2.5
Standard #2	F	2.0	10	1.0, 0.5, 0.5
Standard #1	G	5.0	10.0	0.5, 0.25, 0.25

Source Solution	Concentration (µg/mL)
A = Nitroglycerin	4000
B = PETN Solution	1000
C = Surrogate Stock Solution	1000
D = Standard 5	As Above
E = Standard 4	As Above
F = Standard 3	As Above
G = Standard 2	As Above

2.2.4.4 Note: Store all standard and surrogate solutions in amber glass vials with screw caps and Teflon-lined septa. Minimize headspace in these vials and store them in a refrigerator kept at 4°C ±2°C. Allow standards to come to room temperature prior to use.

2.2.4.5 Stock solutions may be used for up to one year, and working solutions are good for at least six months, or the expiration date of the parent standard, whichever is sooner.

2.2.4.6 All standards are assigned a unique identifier to enable cross-referencing of each individual standard back to the supplier's lot number. In addition, all standards are labeled with the standard concentration, the solvent, date prepared, expiration date, analyst's initials, and the standard reference number. Refer to Laucks' SOP on the traceability, documentation, and preparation standards.

3. Safety precautions

3.1 Routine Safety Precautions

3.1.1 All standards and sample extracts should be handled as if they are hazardous substances.

3.1.2 All compounds analyzed by this method are used either in the manufacture of explosives or are the degradation products of these compounds. When making stock solutions for calibration, treat each explosive compound with caution.

3.1.3 Refer to the instrument manufacturer's manual for routine instrument precautions.

3.1.4 Routine precautions include an awareness of the moving parts on the instrument you're using. These parts are often charged with power from an electrical component or with high pressure gas and have the potential to do harm if not used properly.

3.1.5 Electrical shock - All instruments present the possibility of electrical shock. The operator should take all precautions including ensuring that all instruments are operated with fully grounded power outlets, turning off the instrument and disconnecting the instrument from the electrical power supply before working on any electrical components, etc.

3.1.6 Almost all of the analytes under consideration are known or suspected carcinogens. Analysts should wash their hands after using any standard, solvent or sample extract. Additionally, a respirator should be worn or a fume hood utilized for extremely hazardous compounds or very dirty extracts.

3.2 Waste disposal

3.2.1 Out of date standards and sample extracts are disposed of in the designated organic waste container located in the organic preparation area.

3.2.2 HPLC liquid waste is disposed of by pouring into the designated organic waste container located in the solvent storage area.

4. Operation procedures

4.1 Analytical Conditions

4.1.1 Current C18 Column Conditions for the 14 Compounds:

HPLC3		HPLC4	
Pump A Solvent:	Methanol	Solvent:	Methanol/Water
Pump B Solvent:	Water	Flow:	0-24 min = 0.5 mL/min
Flow:	1.0 mL/minute:		24-35 min = 0.8 mL/min
Gradient:	Linear		35-40 min = 0.5 mL/min
Run Time:	60 minutes	Run Time:	40 minutes
Acquisition Time:	60 minutes	Acq. Time:	35 minutes
Injection Volume:	50 µL inj.	Time:	50 µL

Gradient Profile (HPLC3)		**Run Time Profile**
Initial %A:	40	
Initial %B:	60	
Hold Time:	5 minutes	0 - 5 minutes
Gradient 1 %A:	60	
Gradient 1 %B:	40	
Ramp Time 1:	15 minutes	
Hold Time 1 :	5 minutes	5 - 20 minutes
Gradient 2 %A:	95	
Gradient 2 %B:	05	
Ramp Time 2:	10 minutes	20 - 30 minutes
Hold Time 2:	5 minutes	30 - 35 minutes
Gradient 3 %A:	40	
Gradient 3 %B:	60	
Ramp Time 3:	5 minutes	35 - 40 minutes
Hold Time 3:	20 minutes	40 - 60 minutes (Column Equilibration)

4.1.2 Current CN Column Conditions for the 14 Compounds:

HPLC2 File #6:		
Pump A Solvent:	Methanol	
Pump B Solvent:	Water	
Flow:	1.0 mL/minute	
Gradient:	Linear	
Run Time:	50 minutes	
Acquisition Time:	25 minutes	
Initial %A	25	
Initial %B	75	
Gradient 1 %A:	45	
Gradient 1 %B:	55	
Ramp Time 1:	2 minutes	0 - 2 minutes
Hold Time 1 :	3 minutes	2 - 5 minutes
Gradient 2 %A:	60	
Gradient 2 %B:	40	
Ramp Time 2:	5 minutes	5 - 10 minutes
Hold Time 2 :	8 minutes	10 - 18 minutes
Gradient 3 %A:	25	
Gradient 3 %B:	75	
Ramp Time 3:	7 minutes	18 - 25 minutes
Hold Time 3 :	25 minutes	25 - 50 minutes

4.1.3 Current C18 Column Conditions for the 6 Additional Compounds:

Gradient Profile (HPLC3)		**Run Time Profile**
HPLC3 File:	EXTRAS	
Pump A Solvent:	Water	
Pump B Solvent:	Methanol	
Flow:	1.0 mL/minute	
Gradient:	Linear	
Run Time:	53 minutes	
Acquisition Time:	30 minutes	
Initial %A	90	
Initial %B	10	
Gradient 1 %A:	70	
Gradient 1 %B:	30	
Ramp Time 1:	5 minutes	0 - 5 minutes
Hold Time 1 :	0 minutes	
Gradient 2 %A:	50	
Gradient 2 %B:	50	
Ramp Time 2:	3 minutes	5 - 8 minutes
Hold Time 2 :	7 minutes	8 - 15 minutes
Gradient 3 %A:	26	
Gradient 3 %B:	74	
Ramp Time 3:	2 minutes	15 - 17 minutes
Hold Time 3:	13 minutes	17 - 30 minutes
Gradient 4 %A:	90	
Gradient 4 %B:	10	
Ramp Time 4:	2 minutes	30 - 32 minutes
Hold Time 4:	21 minutes	32 - 53 minutes (Column Equilibration)

4.1.4 Current C8-CN in Series for the 6 Additional Compound Confirmation:

Time	Flow	Methanol	Water
	1.0	15	85
2.4	1.0	15	85
7.0	1.0	40	60
10.0	1.0	50	50
17.0	1.0	50	50
20.0	1.0	80	20
21.0	0	15	85

4.1.5 Current Column Conditions for NG/PETN:

C18		CN	
Pump A Solvent:	Methanol	Pump A Solvent:	Methanol
Pump B Solvent:	Water	Pump B Solvent	Water
Flow:	0.6 mL/minute:	Flow:	1.2 mL/minute
Gradient:	Linear (1:1)	Gradient:	Linear (1:1)
Run Time:	30 minutes	Run Time:	20 minutes
Acquisition Time:	30 minutes	Acq. Time:	20 minutes
Injection Volume:	50 µL inj.	Time:	50 µL

4.2 Method Detection Limit Study

4.2.1 Prior to the analysis of any samples, it is necessary to establish method detection limits. This procedure is fully described in the Laucks SOP on determination of detection limits. Briefly, it involves the analysis of 7 replicate samples spiked at a concentration near the anticipated method detection limit. A Student's T-test is then applied to these measured values to calculate the MDL. MDL studies are performed on both columns.

4.3 Method Validation

4.3.1 Prior to the analysis of any samples, it is necessary to validate the method. A method validation study is performed in a similar manner to an MDL study with the exception that a minimum of 4 replicates are required and the concentration levels are typically higher.

4.4 Retention Time Windows

4.4.1 It is necessary to establish retention time windows for the method by analyzing standards for all target analytes over at least a 24-hour period. A 24-hour time period may be deemed more appropriate for this analysis because the instrument is not always operated for longer periods when analyzing sample extracts. At least 5 standards must be injected during the 24-hour time period, at about equal time intervals. These standards should be interspersed with real sample extracts in order to mimic actual instrument operating conditions. Tabulate the retention times for all standard compounds and compute the sample (n-1) standard deviations of all the retention times.

4.4.2 The retention time window half-width is set at 3 times the above calculated standard deviation. This operation must be repeated whenever major equipment changes are made or whenever the chromatographic method is modified.

4.4.3 In some cases, the retention time window may be modified in order to take into account any pattern shifting. This shift is acknowledged by observation of the surrogate peak behavior and the surrounding CCV standards. If retention time shift has occurred and the possibility of misidentifying peaks exists, then the sample is reanalyzed bracketed by in-control CCV standards.

4.4.4 See the Laucks SOP on determination of retention time windows for more specifics on their determination and use.

4.5 Initial Multi-Point Calibration

4.5.1 Analyze standard solutions (including the surrogate solution) using at least 5 different concentration levels. The lowest concentration defines the reporting limit. The highest concentration should define the upper usable working range of the detector. Inject the standard solutions from the lowest concentration to the highest. Criteria for evaluating these standards are detailed in Section VI.

4.6 Initial Calibration Verification

4.6.1 At the beginning of an analysis sequence, analyze the mid-point calibration standard (STD 3). The computed calibration factor (CF) or concentration measurement must meet the criteria detailed in Section VI.

4.6.2 Since the retention time windows which were established by the retention time study are relative and not absolute, the windows are anchored by the ICV. This allows the retention times from the ICV to become the mid-point of the retention time windows..

4.7 Instrument Blank

4.7.1 After the analysis of Standard 5, an instrument blank (IBLK) is analyzed. This is to verify that there is no carryover between sample injections. Evaluation criteria are detailed in Section VI.

4.7.2 Any sample that is suspected of containing high concentrations of target analytes should be followed by an IBLK. This IBLK analysis is used only to make a judgment as to the possibility of carryover into the sample analysis immediately following the IBLK. Evaluation criteria are detailed in Section VI.

4.8 Continuing Calibration Verification

4.8.1 A mid-range calibration standard (STD 3) is analyzed after every ten sample injections. In addition, this standard must be the last injection made in the analysis sequence. Evaluation criteria are detailed in Section VI.

4.9 Sample Analysis

4.9.1 Analysis sequence

See Appendix II for a detailed analysis injection sequence.

4.9.2 Compound Identification

4.9.2.1 Compounds are tentatively identified if a peak elutes in the retention time window characteristic of that compound on the primary column. To confirm the presence of that compound in the sample extract, the peak must also elute in its retention time window on a second column. There are several analyte co-elutions which occur on the secondary column. In instances where target analytes that have been tentatively identified on the C18 column co-elute on the CN column, positive confirmation can not be made. All analyte quantitations come from the C18 column. The CN column is typically not used for quantitation purposes. The responses of the eluting compounds are summed and reported as one peak. When the presence of these compounds are indicated on the C18 column, CN column concentration values can not be accurately determined due to the coeluting peaks and the sample results are flagged appropriately. Retention time windows are established as previously described and are updated each QC period. Compounds can only be identified if the ICV and CCV criteria detailed in Section VI are strictly adhered to. Due to constraints of the software, the RT of the co-eluting compounds is determined by assigning the RT to the highest point within a give window. For the co-eluting compounds that may have a slight but not complete separation, such as: 2-nitrotoluene, 3-nitrotoluene and 4-nitrotoluene, this determination of RT can result in RT

windows which exceed those identified by the standard (due to the changes in peak heights with concentration values). In order to avoid potential exceedance of RT windows, and in order to accurately identify these co-eluting target analytes, the retention time windows may be administratively set for the co-eluting compounds.

4.9.2.2 The experienced analyst's judgment weighs heavily in evaluating chromatograms for compound identification. For instance, the retention times of surrogate compounds may be outside their expected windows due to sample matrix effects. The analyst may decide to re-adjust the target analyte's retention time windows on an ad hoc basis based on such an observed shift. This can occur only on a sample-specific basis and is used when the analyst examining the data suspects that a retention time shift has occurred. If this is done, it must be fully documented in the case narrative notes. If the concentration of any target analyte exceeds the calibration range, the sample extract must be diluted and reanalyzed.

4.9.3 Compound Quantification

4.9.3.1 Target compound concentrations are calculated using the following equations:

4.9.3.2 Aqueous samples

The external standard equation, as expressed in SW-846 is

$$Concentration(\mu g / L) = \frac{A_x \times A \times V_t \times D}{A_s \times V_i \times V_s}$$

where:

A_x = Response for the analyte in the extract, in area or height units.
A = Amount of standard injected (in nanograms).
A_s = Response for the external standard, same units as A_x.
V_i = Volume of extract injected, µL.
D = Dilution factor of extract. The final result of an algebraic multiplication of the ratio of all dilution final volumes to initial volumes. For example, if an extract is diluted 10 µL to 1000 µL and subsequently diluted an additional 10 µL to 1000 µL, the expression is: (1000/10) * (1000/10) = 100 * 100 = 10,000. If no dilution was made, D = 1.
V_t = Volume of total extract, µL.
V_s = Initial sample size, mL.

4.9.3.3 In routine use at Laucks, the equation reduces as follows.

4.9.3.4 First, CF is used directly in the equation. Since Cf = A_s/A, this substitution is made. Next, since Laucks routinely measures all final extract volumes in mL, a conversion factor for

µL to mL must be made in the numerator of the expression - i.e., V_t = 1000 * mL. Finally, the sample preparation process is represented as the algebraic ratio of initial sample size to final extract volume.

4.9.3.5 The equation then becomes

$$Concentration(\mu g / L) = \frac{1000 \times Ax \times D}{CF \times Vi \times (Vs / Vt)}$$

4.9.3.6 This expression is completely equivalent to the SW-846 equation, yielding the same final result. To report concentrations in alternate units, apply an appropriate factor:

mg/L = µg/L * 0.001

4.9.3.7 Non-aqueous samples

4.9.3.8 The results calculation for non-aqueous samples is very similar to that for aqueous samples. The only difference is the inclusion of a total solids term to calculate the dry weight equivalent of the initial sample size.

$$Concentration(\mu g / kg) = \frac{1000 \times A_x \times D}{CF \times V_i \times (W / V_t) \times T_s}$$

where:

W_s = Sample size extracted in grams.
T_s = Total Solids in decimal format (i.e. 0.76 not 76).

5. Reports

5.1 Data Packet Organization

See Appendix III for a check list detailing data packet organization.

5.2 Quality Control Reports

5.2.1 All results for quality control tests are entered into the lab data base. Printouts of all data entered must be included in the data packet. The routine minimum is a method blank report, a method blank spike report, and an MS/MSD report.

5.3 Data Qualifying Flags

5.3.1 Sample report results are qualified with data qualifying flags. These flags have the following definitions:

U:	The analyte of interest was not detected, to the limit of detection indicated.
B:	The analyte of interest was detected in the method blank associated with the sample, as well as in the sample itself. The B flag is applied without regard to the relative concentrations detected in the blank and sample.
J:	The analyte of interest was detected below the practical quantitation limit. This value should be regarded as an estimate.
D:	The value reported is derived from the analysis of a diluted sample or sample extract.
P:	When a dual column technique is employed, this flag indicates that calculated results from the two columns differ by more than 25 percent.
E:	The value reported is based on a sample or sample extract in which the target analyte concentration exceeded the calibration range. The value reported should be considered an estimate.
X:	The sample has been analyzed at several dilutions. The value reported has been determined to be the most appropriate quantitative value.
Z:	The value reported is from the C18 column only. Due to coelution with another target analyte on the CN column, the compound could not be quantitatively confirmed.

- Due to software constraints, the percent difference will also be calculated when quantitative confirmation is not possible on the confirmation column due to co-elution. When this happens, the concentration value of the confirmation column used to determine the %D is calculated as the sum of the co-eluting compounds and reported as one peak.

6. Quality Control

See the Requirements and Corrective Actions table in Appendix IV for additional information.

6.1 Initial Calibration

6.1.1 Criteria

6.1.1.1 The initial calibration is evaluated by calculating the %RSD of the calibration factors from the five linearity standards.

6.1.1.2 CFs are calculated using the equation:

$$CF = \frac{\text{response}}{\text{ng injected}}$$

6.1.1.3 This %RSD method assumes a linear response with the calibration curve passing through the origin.

6.1.1.4 The calculated CFs are tabulated and the %RSDs calculated. All compounds must have a %RSD of 20% to meet the method criteria.

6.1.2 Corrective action

6.1.2.1 If the %RSD criteria of 20% are not met, the out of control standard should be reanalyzed. If the curve is still out of control, determine the cause of failure and correct. Recalibrate the instrument and reanalyze any samples associated with the out of control curve.

6.2 Initial Calibration Verification

6.2.1 Criteria

6.2.1.1 Using the average CF calculation technique, compute the CFs for each compound. The calibration factors for the ICV standard are compared to the mean CFs for the initial multi-point calibration. The percent difference for these calibration factors is calculated as follows:

$$\%\,Difference = \frac{CF_a - CF_i}{CF_a} \times 100$$

where:
CF_a = .Average CF from the initial multi-point calibration
CF_i = Calibration Factor of the calibration verification standard.

6.2.1.2 The %D cannot exceed 15% for any target analytes or surrogates.

6.2.1.3 The mid-point for the RT window of each compound is updated using the ICV RTs.

6.2.2 Corrective action

6.2.2.1 If the ICV criteria are not met, no sample extracts can be analyzed. Determine the cause of the ICV failure and correct. Reanalyze the ICV and if it is still out of control, a new calibration curve must be analyzed.

6.3 Continuing Calibration Verification

6.3.1 Criteria

6.3.1.1 A CCV standard is analyzed singly for every 10 injections, and after the last sample of the sequence. The CF for each compound is calculated and the percent difference is calculated as shown above.

6.3.1.2 The %D results cannot exceed 15% for any target analytes or surrogates.

6.3.1.3 The retention times for all target analytes must fall within the RT windows established by the ICV.

6.3.2 Corrective action

6.3.2.1 Determine the cause of failure and correct. Reanalyze the calibration curve. All samples bracketed by an out of control CCV must be reanalyzed unless the CCV demonstrates an

increase in response and no analytes are detected above the reporting limit in the associated samples.

6.4 Instrument Blank

6.4.1 Criteria

6.4.1.1 There must be no target analyte levels above the reporting limit in the initial IBLK.

6.4.2 Corrective action

6.4.2.1 If the initial IBLK contains measurable levels of target analytes, the system is out of control. The source of contamination must be identified and corrected.

6.4.2.2 IBLKs are used to monitor for possible carryover in high concentration extracts. Those IBLKs optionally placed into the sequence following suspected high concentration extracts are used to flag the possibility of analyte carryover into the following sample extract. The extract immediately following the out of control IBLK may need to be reanalyzed if there is a detectable amount of the analyte found in the IBLK.

6.5 Method Blank

6.5.1 Criteria

6.5.1.1 Method blanks are used to verify contamination free reagents and apparatus. They are prepared with every set of samples extracted at the same time or at least one blank for every 20 samples, whichever is more frequent. Analytes of interest should not be detected at levels greater than the current reporting limit in the method blank sample. If any analytes are detected above the reporting limit, corrective action must be taken.

6.5.2 Corrective action

6.5.2.1 Reanalyze the blank and check calculations. If it is still out of control, re-extract the entire batch of samples unless the analyte(s) present in the method blank are not present in the associated samples. In any event it is the laboratory's responsibility to ensure that method interferences caused by contaminants in solvents, reagents, glassware, and other sample processing hardware leading to discrete artifacts and/or elevated baselines in the chromatograms be minimized. In the extreme case of chronic contamination, blanks may have to be analyzed from each stage of the sample processing to determine the contamination source so it can be eliminated. In all cases where blank contamination exceeds the control limit a narrative comment must be made which documents the corrective actions taken.

6.6 Blank Spike or QC Check Sample (LCS)

6.6.1 Criteria

6.6.1.1 A blank spike follows the same protocol as the matrix spike analysis except that the spiking solution is added to a method blank solution instead of an actual sample. In addition, this spiking solution is supplied from a source other than the calibration standards. The use of a second source for the spiking standards is employed in order to verify the calibration standards. A method blank with added analytes is a blank spike. Blank spike percent recovery control limits are detailed in the laboratory QC database. These control limits are updated periodically.

6.6.2 Corrective action

6.6.2.1 The blank spike is used to determine whether a method is in control during sample preparation and analysis. Sample re-extraction and reanalysis would be triggered by any analytes falling outside of control limits in the blank spike sample unless all sample surrogate recoveries and MS/MSD spike recoveries are in control.

6.7 Matrix Spike

6.7.1 Criteria

6.7.1.1 A sample is chosen at random (unless designated by project-specific requirements) from the samples to be analyzed, and an aliquot of spiking solution is added to this sample prior to extraction. It is required that a matrix spike analysis be performed with each extraction batch. However, the minimum frequency for MS analysis is 1 each per 20 samples per matrix. This frequency may be changed on a project specific basis. This matrix spike sample is used to evaluate the matrix effect of the sample upon recovery of the analytes. This spiking solution is supplied from a source other than the calibration standards. The use of a second source for the spiking standards is employed in order to verify the calibration standards. The recovery of spike analytes is calculated as follows:

$$\%Recovery = \frac{SSR - SR}{SA} \times 100$$

where:
SSR = Concentration in spiked sample.
SR = Native concentration in unspiked sample.
SA = Concentration of spike added.

6.7.1.2 The current recovery criteria are detailed in the Laucks quality control database. These control limits are updated periodically.

6.7.2 Corrective action

6.7.2.1 Samples with spike recoveries outside control limits will be reviewed for possible corrective action. Corrective action may involve recalculation, re-extraction and/or reanalysis. This process should also look at the recovery of surrogate compounds in the MS sample and at the recovery of matrix spiking compounds from the extraction batch blank spike analysis. In all cases a narrative explanation of the condition is required to detail the corrective actions taken.

6.8 Matrix Spike Duplicate

6.8.1 Criteria

6.8.1.1 The compound recovery criteria are identical to those for the matrix spike sample. In addition, the matrix spike duplicate is used to measure method precision. This is done by computing the relative percent difference (RPD) between the matrix spike and matrix spike duplicate recovery values. This calculation is as follows:

$$RPD = \frac{S1 - S2}{(S1 + S2)/2} \times 100$$

where:
S1 = Measured concentration for MS sample.
S2 = Measured concentration for MSD sample.

6.8.1.2 The current RPD control limits are detailed in the Laucks quality control database. These control limits are updated periodically.

6.8.2 Corrective action

If a trend in out of control RPD values is observed, the methods used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable reproducibility.

6.9 Surrogate Recovery

6.9.1 Criteria

6.9.1.1 Surrogates are chemically similar compounds added to every sample, method blank, and QC sample prior to sample processing. They are used to monitor for potential sample processing errors and matrix effects. Surrogate compound recoveries are calculated as follows:

$$\%Recovery = \frac{S_m}{S_a} \times 100$$

where:
S_m = Concentration of surrogate measured in sample.
S_a = Concentration of surrogate added.

6.9.1.2 Detailed surrogate recovery control limits are detailed in the Laucks quality control database. These control limits are updated periodically.

6.9.2 Corrective Action

6.9.2.1 Reanalyze the sample. Low surrogate recoveries are greater potential indicators of poor method performance than high surrogate recoveries since non-GC/MS methods cannot separate co-eluting interferents. In instances where high surrogate recoveries are attributable to matrix effects, no corrective action is taken. However, if elevated surrogate recoveries are attributable to preparation error, re-extraction and reanalysis is performed.

6.9.2.2 Check calculations for possible error. Re-extract the sample if surrogate recovery is less than the lower control limit. If a poor injection is suspected, reanalyze the sample.

6.9.2.3 Low surrogate recoveries in the method blank may require that all the samples in the associated batch be re-extracted and reanalyzed. In any case, it is imperative to identify the problem associated with low recovery so that it can be corrected. It is a requirement that all out of control surrogate recoveries and the corrective action taken be discussed in the narrative.

7. References

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40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule," October 26, 1984, U.S.E.P.A.

Special Report 93-11, "Experimental Assessment of Analytical Holding Times for Nitroaromatic and Nitroamine Explosives in Soil," Clarence L. Grant et al, June 1993, U.S. Army Cold Regions Research and Engineering Laboratory.

Special Report 93-24, "Evaluation of Pre-extraction Analytical Holding Times for Nitroaromatic and Nitroamine Explosives in Water," Clarence L. Grant et al, August 1993, U.S. Army Cold Regions Research and Engineering Laboratory.

APPENDIX I

Calibration Standard Solution Concentrations, µg/mL

Compound	STD1	STD2	STD3	STD4	STD5
HMX	0.1	0.2	1.0	2.0	10.0
RDX	0.1	0.2	1.0	2.0	10.0
1,3,5-TNB	0.1	0.2	1.0	2.0	10.0
1,3-DNB	0.1	0.2	1.0	2.0	10.0
Tetryl	0.1	0.2	1.0	2.0	10.0
NB	0.1	0.2	1.0	2.0	10.0
2,4,6-TNT	0.1	0.2	1.0	2.0	10.0
4-Am-DNT	0.1	0.2	1.0	2.0	10.0
2-Am-DNT	0.1	0.2	1.0	2.0	10.0
2,4-DNT	0.1	0.2	1.0	2.0	10.0
2,6-DNT	0.1	0.2	1.0	2.0	10.0
2-NT	0.1	0.2	1.0	2.0	10.0
3-NT	0.1	0.2	1.0	2.0	10.0
4-NT	0.1	0.2	1.0	2.0	10.0
Surrogate					
1,2-Dinitrobenzene	0.1	0.2	1.0	2.0	10.0

Calibration Standard Solution Concentrations, µg/mL

Compound	STD1	STD2	STD3	STD4	STD5
TNX	0.1	0.2	1.0	2.0	10.0
2,6-DA-4-NT	0.1	0.2	1.0	2.0	10.0
2,4-DA-4-NT	0.1	0.2	1.0	2.0	10.0
MNX	0.1	0.2	1.0	2.0	10.0
3,5-DNA	0.1	0.2	1.0	2.0	10.0
4,4'-TN-AZOXY	0.1	0.2	1.0	2.0	10.0
Surrogate					
1,2-Dinitrobenzene	0.1	0.2	1.0	2.0	10.0

Calibration Standard Solution Concentrations, µg/mL

Compound	STD1	STD2	STD3	STD4	STD5
NG	0.5	1.0	5.0	10.0	20.0
PETN	0.25	0.5	2.5	5.0	10.0
Surrogate					
1,2-Dinitrobenzene	0.25	0.5	2.5	5.0	10.0

Surrogate Stock Solution

Compound	Concentration
1,2-Dinitrobenzene	1000 µg/mL

IBLK Solution

Compound	Concentration
1,2-Dinitrobenzene	2.0 µg/mL

APPENDIX II

Analysis Sequence

Injection	Sample
1	Standard #1
2	Standard #2
3	Standard #3 (ICV Standard)
4	Standard #4
5	Standard #5
6	IBLK
7	ICV
	injections (8-16)
17	IBLK
18	CCV Standard
19	injections (19-27)
28	BLK
29	CCV Standard
	injections (total of 10 injections between CCVs)
last	IBLK
last	CCV Standard

APPENDIX III

Data Packet Order List

I. QC SUMMARY

Surrogate Recovery Summary Report
Blank Spike Report
MS/MSD Report
Method Blank Summary

II. SAMPLE DATA:

Organic Analysis Data Sheet
Sample Confirmation Worksheet
Chromatogram, primary column
Quantitation Report, primary column
Chromatogram, secondary column
Quantitation Report, secondary column

III. STANDARD DATA:

Linearity Report
ICAL Data
ICAL Response, ICAL std concentrations
ICV reports
CCV reports
Other Standards Used to Support Sample Data and Instrument Blanks

V. Raw QC Data:

Method Blank
Chromatograms
Quantitation Report

Blank Spike
Chromatograms,
Quantitation Report

Matrix Spike
Chromatograms
Quantitation Report

Matrix Spike Duplicate
Chromatograms
Quantitation Report

V. Bench Sheets

SDG Report
Extraction Bench Sheets
Injection Sequence (logbook copy)
Miscellaneous Work Sheets. (%TS, calcs, HTVRs)
Standards Logs

VI. Reject Data:

Data not used to support reported sample results.
All data acquired but rejected on account of QC out of control.
Non-routine standards used to support sample data should be placed at the last of the Standard Data section.

APPENDIX IV

Method 8330 Requirements and Corrective Actions					
QA Element	**Method Criterion**	**Laucks Criterion**	**Frequency**	**Corrective Action**	**Documentation**
Initial Calibration	Minimum of 5 levels.	Minimum of 5 levels. Must demonstrate an RSD of <20%.	As necessary due to major instrument maintenance or difficulties meeting the CCV requirements.	Reanalyze out of control standard. If still out, determine cause of curve failure and correct. Re-analyze curve and any samples analyzed against curve.	Target forms and raw data.
Initial Calibration Verification	+/- 15% of the initial calibration response factor.	+/- 15% of the initial calibration response factor.	Every 24 hr. or at the beginning of an analytical sequence, which ever is more frequent, or as necessary.	Determine cause of ICV failure and correct. Reanalyze ICV and if out of control a new calibration curve must be analyzed.	Target form and raw data.
Continuing Calibration Verification	+/- 15% of the initial calibration response factor. Analyze singly every 10 injections and after the final sample.	+/- 15% of the initial calibration response factor.	Every 10 injections and at the end of an analytical sequence.	Determine cause of failure and correct. Reanalyze calibration curve. All samples bracketed by an out of control CCV must be reanalyzed unless the CCV demonstrates an increase in response and no analytes are detected above the reporting limit.	Target form and raw data.
Instrument Blank	Analyze after analysis of a sample with analyte levels which exceed the upper calibration level.	Analyze after samples when high levels of matrix are suspected.	As necessary.	Reanalyze any samples with suspected carryover.	Raw data
Method Blank	All analytes must be < MDL	All analytes must be less than the Reporting Limit	One method blank per 20 samples or each extraction batch, whichever is more frequent.	Reanalyze blank. If still out of control, re-extract the entire batch of samples unless the detected analyte(s) are not present in the associated samples.	Method Blank Summary and raw data. Narrative comment when necessary.

Method 8330 Requirements and Corrective Actions					
QA Element	**Method Criterion**	**Laucks Criterion**	**Frequency**	**Corrective Action**	**Documentation**
Surrogate Recovery	Minimum of 1 surrogate. Recoveries must fall within the labs established windows.	See latest version of Laucks Testing Labs control limits catalogue. All surrogate recoveries must fall within the generated limits. If laboratory limits are not established due to the implementation of a new surrogate , the default limits of 70% - 130% are used until enough data points are collected to generate new limits.	All samples, method blanks, and QC samples.	Re-extract if surrogate recovery is < the lower control limit. If a poor injection is suspected reanalyze the sample.	Target Surrogate Summary form or special test results. Narrative comment when necessary.
MS/MSD	One per 20 samples or extraction batch which ever is more frequent. Must be per matrix.	See latest version of Laucks control limits located in the QC database.	1 MS/MSD pair per 20 samples or every extraction batch which ever is more frequent.	Per SW 846, if analyte recoveries are out of control in the MS/MSD but are in control in the associated blank spike, no further action is required.	MS/MSD report and raw data. Narrative comment when necessary.
Blank Spike	N/A	See latest version of Laucks Testing Labs control limits in the QC database. All recoveries must fall within the generated limits.	1 per 20 samples or every extraction batch which ever is more frequent.	Re-extract if analyte recoveries are outside of control limits.	Blank Spike report and raw data. Narrative comment when necessary.

APPENDIX V

Elution Order of Target Analytes

Compound	Order of Elution on Primary (C18) Column	Order of Elution on Confirmation (CN) Column
HMX	1	10
RDX	2	8
1,3,5-TNB*	3	1
1,3-DNB	5	2
Tetryl	6	9
NB*	7	1
2,4,6-TNT*	8	4
4-Am-DNT	9	6
2-Am-DNT	10	7
2,4-DNT*	11	4
2,6-DNT*	12	4
2-NT*	13	3
3-NT*	14	3
4-NT*	15	3
Surrogate		
1,2-Dinitrobenzene	4	5

Elution Order of 6 Additional Analytes

Compound	Order of Elution on Primary (C18) Column	Order of Elution on Confirmation (C8+CN) Column
TNX	1	1
2,6-DA-4-NT	2	2
2,4-DA-6-NT	3	3
MNX	4	4
3,5-DNA	6	6
4,4'-TN-AZOXY	7	7
Surrogate		
1,2-Dinitrobenzene	5	5

Elution Order of PETN/NG

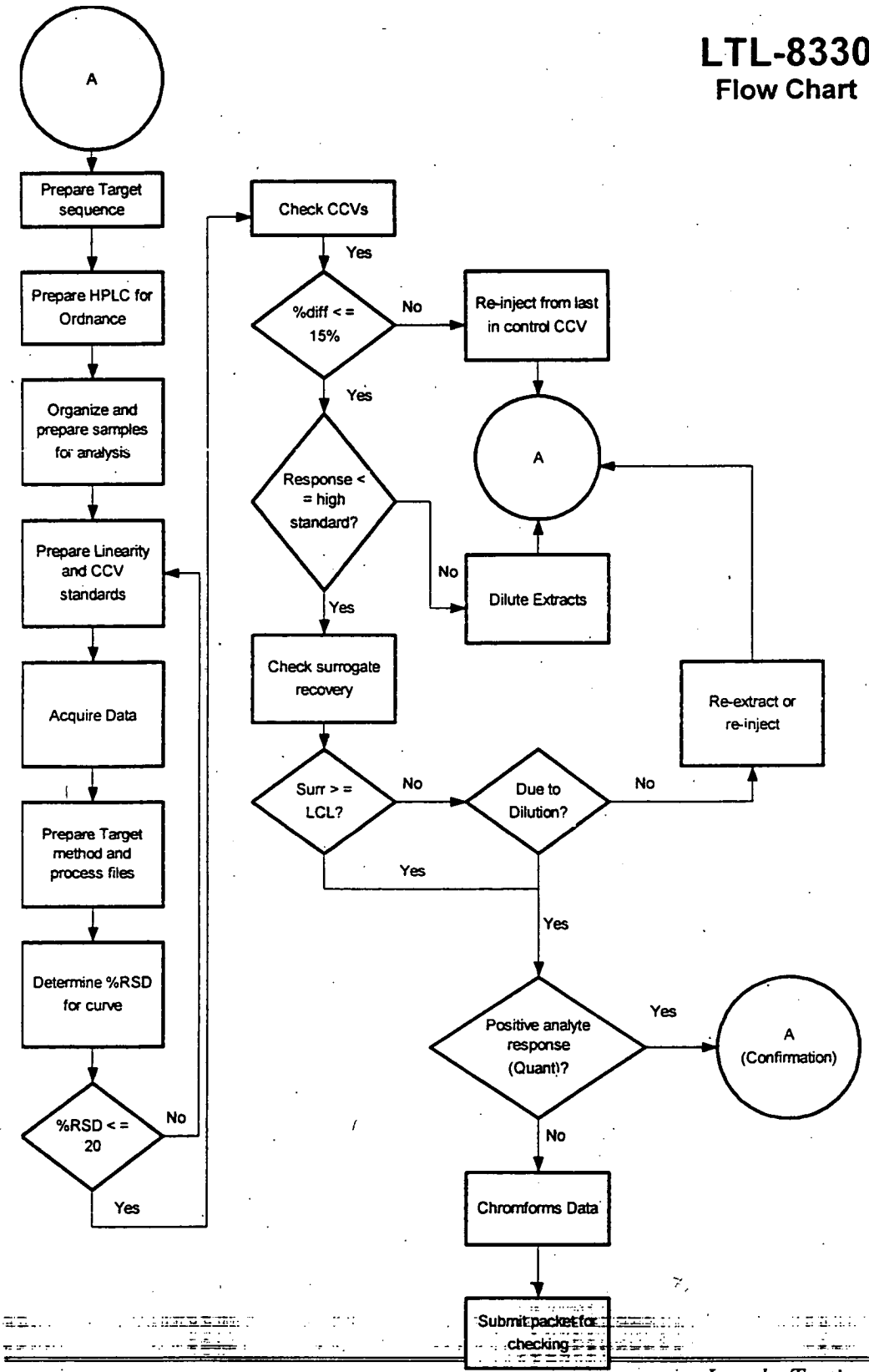
Compound	Order of Elution on Primary (C18) Column	Order of Elution on Confirmation (CN) Column
NG	2	2
PETN	3	3
Surrogate		
1,2-Dinitrobenzene	1	1

*Indicates the compound co-elutes on the confirmation (CN) column.

APPENDIX VI

Method 8330 Flow Chart

LTL-8330
Flow Chart



APPENDIX II

Analysis Sequence

Injection	Sample
1	Standard #1
2	Standard #2
3	Standard #3 (ICV Standard)
4	Standard #4
5	Standard #5
6	IBLK
7	ICV
	injections (8-16)
	17 IBLK
	18 CCV Standard
	19 injections (19-27)
	28 BLK
	29 CCV Standard
	injections (total of 10 injections between CCVs)
last	IBLK
last	CCV Standard

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP : LTL-9104

Title: **Preparation and Analysis of Total and Amenable Cyanide according to EPA Methods 335.1, 335.3, and SW 846 method 9012A.**

Revision history:

Number	Date
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1	02/07/96

Revised by: Bill Lundberg, Division Manager — Date: 2/9/98

Reviewed by: Harry Romberg, QC Officer — Date: 2-9-98

Approved by: Karen Kotz, Laboratory Director — Date: 2/9/98

UNCONTROLLED

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1. Introduction and Scope

1.1 Method Description

Cyanide in samples is determined by a preparation with manual distillation followed by an automated spectrophotometric analysis on an auto analyzer with a UV digester and in-line distillation. The routine reporting limit is 10 µg/L in waters, and 0.50 mg/kg DB in soils and sediments. Pretreatment for the determination of cyanides amenable to chlorination in waters is also included in this SOP.

This SOP is applicable to the sample preparation (for cyanide analysis) of drinking waters, surface and saline waters, domestic and industrial wastes according to EPA methods 335.1, 335.3 and SW 846 method 9012A with the main difference being the use of the in-line still and UV digestion in EPA 335.3. Also, MIDI distillation with a 50 mL water sample instead of the normal distillation of a 500 mL sample is used in this SOP. Other differences from 9012A are listed on the QA chart at the end of this SOP. Soils and sediments are MIDI distilled as a 1 g sample.

This method is restricted to use by, or under the supervision of analysts experienced in the technique described and in SW 846 methodology. Each analyst performing this method must have demonstrated the ability to perform the described analysis.

1.2 Method Deviations and Comments

The following items represent deviations of Method 9012A, as published, which are followed as standard operating procedures in the performance of this method at Laucks:

- The MIDI distillation procedure is used, exactly as written in the CLP statement of work. This distillation uses a 50 mL aliquot of sample and a final distillation volume of 50 mls. This procedure allows the lab to use less sample, and dispose of smaller volumes of waste material. All molar ratios in this distillation are identical to the method as written.

- The calibration curve specified in 9012A is reduced by 2 to compensate for the lack of sample concentration in the distillation step.

1.3 Method Summary

Sample Preparation: The MIDI distillation procedure applies to relatively solid samples such as soils, sediments and sludges, or if requested to water samples. Cyanide determined by this method is defined as cyanide ion and complex cyanides converted to hydrocyanic acid by

reaction in a reflux system with mineral acid in the presence of magnesium ion. The cyanide as hydrocyanic acid (HCN) is released from cyanide complexes by means of a MIDI reflux-distillation operation and absorbed in a scrubber containing sodium hydroxide solution. The cyanide in the distillate is then determined in a spectrophotometric analysis.

Water samples analyzed under EPA 335.3 are not externally distilled and are directly analyzed unless otherwise requested.

Spectrophotometric Analysis: On a Technicon auto analyzer, the samples are acidified and passed through a UV digester and then distilled in-line. The cyanide released is converted to cyanogen chloride, CNCl, by reaction with chloramine-T at pH less than 8 without hydrolysis to the cyanate. After the reaction is complete, color is formed on the addition of pyridine-barbituric acid reagent. The absorbance is read at 570 nm. To obtain colors of comparable intensity, it is essential to have the same salt content in both the samples and the standards.

1.4 Sample Handling, Sample Storage, Holding Times

Samples are preserved with 8 pellets of sodium hydroxide per liter of sample (pH > 12) at the time of collection. Samples must be stored at 4°C ± 2°C and must be analyzed within the specified holding time of 14 days from date of collection. The samples can be submitted in either plastic or glass containers. The pH is checked at the time of analysis and the client should be notified if the pH is less than 12.

Samples are to be checked for chlorine interference at the time of collection and the appropriate action should be taken (see section 5.4 on interferences).

1.5 Definition of Terms

This section defines terms and acronyms as they are used in this SOP. Other terms are not defined here since it is assumed that the user of this SOP already understands their more general meaning.

Batch Identifier - A number given to each preparation or analysis group which uniquely identifies that batch. This number is generally the blank ID for preparation batches only preceded by an "A" rather than a "B" for inorganic batches. The preparation batch IDs are discussed in other documentation. The batch identifier for the second set of water samples analyzed on Feb 6, 1998 would be A020698AA1W02.

CCB - Continuing Calibration Blank - This is the same acronym used in the CLP program. This is a blank which is analyzed immediately after the CCV (almost always after every 10 samples and at the end of the analytical run) during the analysis sequence to determine whether the instrument or system has maintained a stable baseline.

CCV - Continuing Calibration Verification. - This is the same acronym used in the CLP program. This is a standard analyzed at some prescribed frequency (almost always after every 10 samples and at the end of the analytical run) during the analysis sequence to determine whether the instrument or system has remained in calibration.

CLP - Contract Laboratory Program - The USEPA program that contracts with laboratories to provide laboratory services. The term has come to mean a much broader set of methods and deliverables. In context of this SOP, CLP means procedures or operations which are detailed in the CLP contract and which are extended to a broader working definition.

Corr Coef, CC, r - Correlation coefficient - A measure of the "goodness of fit" of a set of data to a regression model. The closer the value is to 1, the higher the degree of confidence in the correlation.

DIW - Deionized water - Lab reagent water. This water should be free of virtually all analytes.

ICB - Initial calibration blank - This term is borrowed from CLP. An instrument blank is made up in the same way as calibration standards, without target analytes.

ICV - Initial calibration verification - This term is borrowed from the CLP protocol. It is a standard which is analyzed at the start of each analytical run and compared to the initial calibration to determine whether the instrument calibration is accurate.

MDL - Method detection limit - The lowest concentration a sample which will yield a positive result that is greater zero at a known level of confidence. MDLs are empirically determined by Laucks.

SRM or LCS - Standard Reference Material or Laboratory Control Sample. This is a material of approximately the same matrix as the samples, containing a known and usually certified amount of target analyte and which is prepared and analyzed in the same manner as a typical sample. This sample is used to demonstrate that the analytical system is in control.

QC period - Quality control period - An analysis sequence initiated by the analysis of one or more standards, followed by samples, and terminated with a standard and blank analysis. A QC period can be open-ended chronologically, but calibration verification must be documented using the procedures in this SOP

Sequence - A set of sample extracts and standard solutions injected into an instrument in a chronologically continuous group. See also QC period.

2. Equipment, Reagents, and Standards

2.1 Equipment

Required instrumentation includes the following:

1) Midi reflux distillation system with heating block capable of maintaining 125°C ± 5°C. We use the MIDI-VAP model MC-100 ("CYN-TEN").

2) Technicon Auto-Analyzer with in-line distillation unit and UV digester.

2.2 Reagents

1) 0.25 N NaOH: dissolve 10.0 g NaOH pellets in 1 L DIW. **CAUTION**: this reagent will get hot upon mixing!

2) $MgCl_2$ solution: dissolve 510 g $MgCl_2 \cdot 6H_2O$ and dilute to 1 L with DIW.

3) 1:1 H_2SO_4: carefully and slowly add with mixing 500 mL of conc. H_2SO_4 to 500 mL of DIW. **CAUTION**: this reagent will get **EXTREMELY** hot upon mixing! Mix in a Pyrex or equivalent container! **DO NOT ADD WATER TO ACID!**

4) Phosphate buffer: dissolve 13.6 g monobasic potassium phosphate (KH_2PO_4) and 0.28 g anhydrous dibasic sodium phosphate (Na_2HPO_4) and dilute to 1 L with DIW.

5) Chloramine -T solution: dissolve 1.0 g in 250 mL of DIW. Prepare this reagent fresh daily.

6) Color reagent: place 15 g of barbituric acid in the bottom of a container, and rinse down the sides of the container with DIW. Add 100 mL of DIW and swirl gently. Add 75 mL of pyridine and mix. Add 15 mL of conc. HCl, mix, and cool. Dilute to 1 L with DIW. This reagent should be stored at 4°C. This reagent should be made under a hood.

7) Distillation reagent: to 500 mL of DIW add 250 mL conc. H_3PO_4 and 50 mL 50% H_3PO_2 and then dilute to 1 L with DIW.

8) Sodium Hypochlorite solution: Commercial bleach such as Clorox (5.5%).

2.3 Standards

(a) Stock cyanide solution for standards and spiking, 100 mg/L CN. Place 0.2510 g of KCN and approx. 1.5 g NaOH in a 1.0 L volumetric flask and dissolve. Dilute up to the mark with DIW. Record this standard in a standards logbook. The stock standard is good for one year.

(b) Intermediate cyanide standard solution, 1.00 mg/L CN. Dilute 1.0 mL of stock standard solution (a) to 100 mL with 0.25 N NaOH. Prepare fresh solution at least weekly and record in a standards logbook.

(c) Blank solution for zero standard, blanks and diluting samples and standards: Dilute 200 µL 10 N NaOH to 100 mL with DIW. Prepare fresh solution at least weekly and as needed.

(d) Calibration standards:

Concentration (mg/L)	µLs of (b)	Final volume (mL)
0.200	1000	5.0
0.150	750	5.0
0.100	500.	5.0
0.050	250.	5.0
0.020	100	5.0
0.010	50.	5.0
0	0	5.0

All standards should be diluted up to final volume with (c) Blank solution. Standards are used up in analysis so prepare fresh standards for each analytical run.

3. Safety Precautions

Refer to the Laucks SOP on lab safety practices and the instrument manufacturer's manual for routine instrument precautions.

All standards, samples and sample solutions should be handled as if they are hazardous substances. Some forms of cyanide can be released as a gaseous form upon acidification. Treat all samples as if they definitely contain cyanide.

For autoanalyzed water samples, a PBW is analyzed for every 20 samples (or with each SDG) analyzed. This does not include distillates from manual distillation which are represented by their distilled PB. This consists of a sample of blank solution (standard (c). If the cyanide concentration is above the reporting limit, then the blank solution is reprepared and reanalyzed as well as any samples diluted with the solution since the last PBW was checked (including the calibration). If the new PBW is in control, then the samples are verified by the new PBW. If contamination persists, then source isolated and the instrument is recalibrated and all samples are verified by the PBW are reanalyzed.

4.7 Matrix Spike

For manual distillation preparation, a sample is chosen at random from the samples to be analyzed (or a sample that is specifically requested by the client if they send excess sample volume). 50 µL of 100 mg/L CN standard solution, standard (a), is added to the 50 mL of sample prior to distillation, for a spike value of 0.100 mg/L in the 50 mL of distillate. For 9012A a 50 mL aliquot is spiked with 20 µL for a spike concentration of 0.040 mg/L The analyst must 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 matrix spike sample is used to evaluate the matrix effect of the sample upon recovery of the analytes.

For autoanalyzed water samples, the same criteria for sample selection and frequency applies. The MS is prepared by measuring out 10 mL of the selected sample, then adding 10 µL of the 100 mg/L CN standard (standard (a)) for a spike value of 0.100 mg/L. If the sample needs dilution, the diluted sample is spiked in the same manner for a spike value of 0.100 (multiplied by the dilution factor) mg/L.

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

If the spike recovery is not at or within the limits stated in QC-DB, an action such as re-analysis of the spike at a higher dilution, or selection of an alternate sample for spiking (if possible) is

taken. An exception to this rule is granted in situations where the blank spike is in control, or the sample concentration exceeds the spike level by a factor of four or more. In these cases, a narration is included with the data.

4.8 Matrix Spike Duplicate

For both manual distillation and autoanalysis; at least one duplicate sample per 20 samples per matrix is required. RPD values are calculated as follows:

$$RPD = \frac{|SR1 - SR2|}{(SR1 + SR2)/2} * 100$$

where:
SR1 = measured concentration in the initial spike analysis
SR2 = measured concentration in the duplicate spike analysis

The recovery and RPD must fall within the control limit stated in QC-DB.

4.9 Blank Spike

To verify the manual distillation procedure, a 50 mL sample of DIW is spiked with 50 µL of the 100 mg/L standard (standard (a)) for a spike value of 0.100 mg/L, and is distilled and analyzed with the other samples. The calculated recovery must be within the limits stated in QC-DB. The minimum frequency for the BS is one per 20 samples per analytical run.

4.10 Sample Duplicate

4.10.1.1 Sample duplicates are required. At least one 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:

$$RPD = \frac{|S1 - S2| * 100}{(S1 + S2)/2}$$

where:
S1 = measured concentration in the initial analysis
S2 = measured concentration in the duplicate analysis

4.10.1.2 The RPD control limits are detailed in the current Control Limits Catalog and in the Quality Control Database (QC_DB) and will change from time to time.

4.10.2 Corrective action

4.10.2.1 If a trend in out of control RPD values is observed, the methods used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable reproducibility. Generally, if recoveries are in control and no analyte of interest was detected in any of the samples, no immediate action will be taken on that sample set. If integrity of reported sample values is in doubt, re-analysis may be called for. Corrective actions should be discussed with the Quality Control Officer.

5. Operation procedures

5.1 Pre-distillation Sample Screen for Waters

Water samples are screened for sulfide and chlorine interferences prior to distillation as follows:

1) Pour a small amount of pH 4 buffer into a 1oz. plastic cup.

2) Check for the presence of sulfide with lead acetate test paper. Wet the paper with pH 4 buffer and then drop ~10 µL of sample onto it. A darkening of the paper indicates the presence of S^{2-}. If present then see Section 5.3 Interferences before proceeding with the distillation.

3) Check for the presence of chlorine with potassium iodide-starch test paper. Wet the paper with pH 4 buffer and then drop ~10 µL of sample onto it. A bluish discoloration indicates the presence of chlorine. If present then see Section 5.3 Interferences before proceeding with the distillation.

5.2 Pretreatment for Cyanides Amenable to Chlorination

Two sample aliquots are required for chlorine amenable cyanide determination. One is pretreated, the other is not pretreated. Both are prepped and analyzed and the difference between the two is the cyanide amenable to chlorination.

1.) To the sample aliquot designated for pretreatment add bleach solution dropwise until an excess of chlorine is maintained. Test for residual chlorine with KI-starch paper. Paper will turn blue.

2.) Maintain excess for one hour with continued agitation using a Teflon stir bar and a stir plate.

3.) After one hour, add ascorbic acid in small increments (~0.01 g) until KI-starch paper shows no residual chlorine and analyze.

5.3 Manual Distillation

Samples are distilled using the MIDI distillation method. The distillation procedure is as follows:

1) Remove fritted tubes from absorbers, rinse, and set aside.

2) Rinse out absorbers, and invert to drain.

3) Add 50.0 mL of a liquid sample (or 1.0 g of a solid sample) to a sample tube. For solid samples, dilute with DIW to the 50 mL mark on the tube.

4) Set up sample tubes for the appropriate QC (BS, PB, MS/MSD), but do not spike the BS or the "spiked" sample yet.

5) Add 3-4 boiling stones to each tube.

6) Insert reflux impingers into the sample tubes.

7) Fill the absorber tubes with 50.0 mL of 0.25 N NaOH, and insert the fritted tubes.

8) Place sample tubes in the back row of the heating block, and the absorbers into the front row.

9) Insert the cold-finger condensers into the top of the sample tubes.

10) Hook up all vacuum hoses.

11) Turn on water flow, and adjust flow meter to approx. the same number of samples that are being distilled (about 9-10 for 10 samples).

12) Plug in the vacuum pump.

13) Stabilize the flow rate at about 3 bubbles per second and wait for about 5 minutes.

14) Spike the BS and the MS/MSD with the appropriate spiking material.

15) Add 5 mL of 1:1 H_2SO_4 to each tube, and let mix for about 3 minutes.

16) Add 2 mL $MgCl_2$ to each tube, and rinse tubes with DIW.

17) Turn power on, and turn knob to mark.

18) When distillation is complete (10 minutes to heat to reflux, 60 mins of reflux, and 15 minutes of cooling) the heating block will automatically shut itself off. Replace tubes in tube rack, removing the fritted tubes before turning the vacuum pump off.

19) Pour off the liquid from the absorbers into a clean container, label, and store at 4°C if the distillates will not be analyzed immediately.

20) Rinse all glassware with DIW, and store fritted tubes in DIW.

5.4 Interferences

1.) Sulfide interference: Some sulfides convert CN^- to SCN^-. Add cadmium carbonate to sample until lead acetate test paper no longer darkens (treat 25 mL more than is necessary for the distillation). Yellow cadmium sulfide precipitates if the sample contains sulfide. Avoid a large excess of cadmium carbonate and a long contact time in order to minimize a loss by complexation or occlusion of cyanide on the precipitated material. Filter sample through a dry filter paper into a dry container then proceed with the distillation procedure. A narration is included in the final report. Run a standard or BS treated in the same manner with the treated samples.

2.) Chlorine interference: Chlorine decomposes most cyanides. Add ascorbic acid to the 50 mL sample ~0.02 g at a time until KI-starch test paper no longer turns bluish then add an additional 0.02 g of ascorbic acid. Proceed with the distillation procedure. A narration is included in the final report.

3.) NO_3 / NO_2 interference: Nitrates and/or nitrites can form oximes with organic compounds which can decompose under test conditions to form HCN. If high levels are suspected (e.g. fertilizers) then sulfamic acid is added to the sample to remove the interference.

4.) Surfactants. For manual distillation, the presence of surfactants may cause the sample to foam during refluxing. If this occurs, add an antifoam agent such as Baker Antifoam B Silicone Emulsion.

5.) Interferences are eliminated or reduced by the distillation procedure

6. Sample Analysis Using a Technicon Auto Analyzer

6.1 Instrument start-up and operation

The auto-analyzer should be set up for cyanide analysis according to the Laucks SOP on configuring the Technicon Autoanalyzer. Turn on power and allow heating bath to reach 150 degrees C while flushing reagent tubes with DIW. After the temperature is reached, place the reagent tubes into their proper reagents. Turn on chart drive motor and allow reagents to flow through completely. When baseline has settled, set baseline at about 90%T.

6.2 Analysis procedure

1) Prepare the 1.0 ppm working standard and the blank solution

2) From these solutions make the standard curve in 1 oz plastic cups, swirl and transfer to sampler cups, and load onto sampler.

3) Immediately after the calibration standards, load an EPA approved LCSW as the ICV. Immediately after the ICV, load the blank solution for the ICB.

4) Load standards, water samples, and distillates from manual distillation (distillates are analyzed the same as water samples) as in the following example and press the reset button on the autosampler to begin the analysis:

Appendix I
Laucks Testing Laboratories
Method LTL 9104 QA Requirements and Corrective Actions

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentatio
Initial Calibration	7 standards plus a blank. r ≥0.995	7 stds plus a blank, one std distilled as the BS. r ≥0.995	Beginning of run	Recalibrate if r < 0.995	In raw data.
Initial Calibration Verification / LCSW	Verify calibration every 15 samples with an independently prepared check standard.±15%.	Independent source ±15%.	1 per batch or 5% including one immediately after calibration.	Recalibrate	Enter in QC database under SRM.
Initial Calibration Blank	No criteria.	≤0.010 mg/L	1 per run immediately after ICV.	Recalibrate	In raw data.
Continuing Calibration Verification	Verify calibration with the mid-range standard. Must agree within 15% of true value.	Verify calibration with the mid-range standard. Must agree within 15% of true value.	1 after every 10 samples, and at the end of the run.	Perform instrument maintenance and recalibrate.	In raw data.
Continuing Calibration Blank	No criteria.	≤0.010 mg/L or reporting limit	1 after every 10 samples, and at the end of the run.	Check for source of contamination. Reanalyze associated samples.	In raw data.
Preparation Blank Soil: Water:	No criteria. No criteria.	≤0.50 mg/kg DB ≤0.010 mg/L or reporting limit	1 per batch or 5%.	Check for source of contamination. Reanalyze / reprep w/ associated samples.	Enter in QC database under BLANKS.
Matrix Spike (%Rec) Soil: Water:	Run one spike duplicate every 10 samples. No recovery criteria .	Refer to current control limits catalog	1 per batch or 5%	Spike at higher level or choose another sample and reanalyze / reprep w/ associated samples.	Enter in QC database under MSPKDATA.
Matrix Spike Dup. (RPD) Soil: Water:	See MS.	Refer to current control limits catalog	1 per batch or 5%	Same as MS.	Same as MS.
Blank Spike (%Rec) (Manually distilled soils and waters only.)	Two stds distilled and compared to equal values in standard curve.	Refer to current control limits catalog	1 per batch or 5%	Reprep w/ associated samples.	Enter in QC database under BS_CALC.

Appendix II

Excel spreadsheet used for cyanide calculations.
(example)

Cyanide Analysis	ITEM	DATE	METHOD	ANALYST	CHECKER
JOB#:	prep.		Man. Distill	PL	
MATRIX: water	analysis	1/25/96	EPA 335.3	PL	

STANDARD CURVE LCSW: WP586 (tv = 0.500 ppm)

standard stock:
TE4-67-17

T.V. (mg/L CN)	Peak Ht.	Baseline	%T	Absorbance
0.000	94.00	94.50	99.47	0.002
0.010	88.50	94.50	93.65	0.028
0.020	80.50	94.50	83.44	0.055
0.050	67.50	94.50	71.43	0.146
0.100	47.50	94.50	50.26	0.299
0.150	30.50	94.50	36.24	0.512
0.200	17.75	94.50	18.78	0.726
--x value--				--y value--

r = 0.9999 m = 2.9043 y(0) = 0.0022 x(0) = -0.0008

ms/msd = 0.100ppm

Sample I.D.	Dilution	Peak Ht.	Baseline	%T	Absorbance	CN (mg/L)	
ICV / LCSW1	5	54.25	95.00	57.11	0.243	0.4151	83%
ICB	1	94.25	95.00	99.21	0.003	0.0004	
MDL tv = 0.005	1	91.25	95.00	96.05	0.017	0.0053	105%
PBW1	1	94.50	95.00	99.47	0.002	0.0000	
5555-1	1	94.50	95.00	99.47	0.002	0.0000	
5555-1ms	1	48.00	95.00	50.53	0.296	0.1013	101%
5555-1msd	1	48.25	95.00	50.79	0.294	0.1005	101%
5555-2	1	94.25	95.00	99.21	0.003	0.0004	
5555-3	1	92.00	95.00	96.84	0.014	0.0040	
5555-4	1	89.00	95.00	93.68	0.028	0.0090	
5555-5	1	88.50	95.00	93.16	0.031	0.0098	
5555-6	1	65.75	95.00	69.21	0.160	0.0543	
CCV1 tv = 0.100	1	45.50	95.00	47.89	0.320	0.1093	109%
CCB1	1	94.00	95.00	98.95	0.005	0.0008	

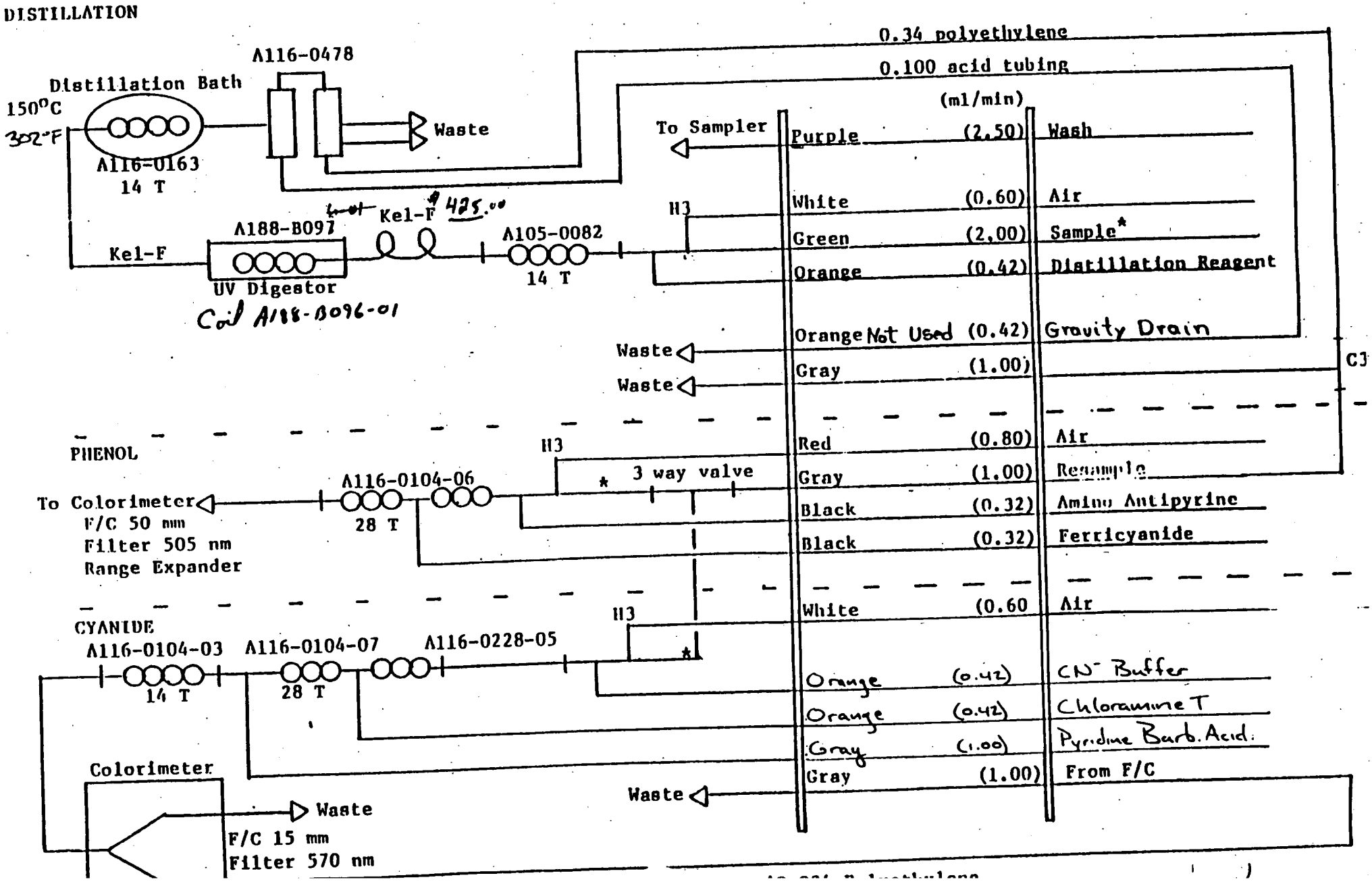
Appendix III

Autoanalyzer Flow Diagram

[illegible] chemistry
currently in use.
Jan 29, 1981 MS

CYANI[illegible]NOL
Combined Manifold
AAI

ALPKEM Corporation
1981
RRP



LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP No. **LTL-9108**

Previous SOP No. **LX-0008-1**

Title: **Phosphorus Analysis, All Forms**

Rev : 1

Laucks is in the process of re-numbering our SOPs. As an interim measure, this page serves as the cover page for those SOPs whose header information has not been updated. This page details the title, the SOP number that it is being controlled under, and the previous SOP number. The previous SOP cover sheet has been manually corrected to reflect the change but each page header will reflect the old numbering system. As SOPs are revised, the full header and cover page will be updated.

UNCONTROLLED

Phosphorus Analysis, all forms

Method# ~~TX-0008-1~~ 12/22/95
LTL-9108

April 21st, 1988

These methods are applicable to the determination of specified forms of phosphorus in drinking, surface and saline waters, domestic and industrial wastes. The methods are based on reactions that are specific for the orthophosphate ion. Thus, depending on the prescribed pre-treatment of the sample, various forms of phosphorus may be determined. These forms are defined in section 1.

Submitted by: Kirk R. Hintzen — Date: 04/22/88
Kirk R Hintzen

Reviewed by: Mike Nelson — Date: 5/13/88
Mike Nelson

Approved by: Jim Owens — Date: June 1, 1988
Jim Owens

1. DEFINITIONS

NOTE: Most common "in house" acronym appears in ( ). If ( ) are empty then that form is not routinely analyzed.

1.1 Total Phosphorus (T-P): All of the phosphorus present in the sample, regardless of form, as measured by the persulfate digestion procedure.

1.1.1 Total Orthophosphate (O-P): Inorganic phosphorus [$(PO_4)^{-3}$] in the sample as measured by the direct colorimetric analysis procedure.

1.1.2 Total Hydrolyzable Phosphorus ( ): Phosphorus in the sample as measured by the sulfuric acid hydrolysis precedure, minus pre-determined orthophosphates. This hydrolyzable phosphorus includes polyphosphorus [$(P_2O_7)^{-4}$,$(P_3O_{10})^{-5}$, etc.] plus some organic phosphorus.

1.1.3 Total Organic Phosphorus ( ): Phosphorus (inorganic plus oxidizable organic) in the sample as measured by the persulfate digestion procedure, and minus hydrolyzable phosphorus and orthophosphate.

1.2 Dissolved Phosphorus (D-T-P): All of the phosphorus present in the filtrate of a sample (filtered through a phosphorus-free filter of .45 micron pore size) as measured by the persulfate digestion procedure.

1.2.1 Dissolved Orthophosphate (D-O-P): Inorganic phosphorus [$(PO_4)^{-3}$] in the filtrate of a sample as measured by the direct colorimetric analysis procedure.

1.2.2 Dissolved Hydrolyzable Phosphorus ( ): Phosphorus in the filtrate of a sample as measured by the sulfuric acid hydrolysis precedure, minus pre-determined dissolved orthophosphates.

1.2.3 Dissolved Organic Phosphorus ( ): Phosphorus (inorganic plus oxidizable organic) in the filtrate of a sample as measured by the persulfate digestion procedure, and minus dissolved hydrolyzable phosphorus and orthophosphate.

1.3 Insoluble Phosphorus ( ): If sufficient amounts of phosphorus are present in the sample, this form may be calculated. i.e. (T-P) minus (D-T-P), (O-P) minus (D-O-P), etc.

2. SAFETY

2.1 Always use safe laboratory procedures.

2.2 Special care should be used when working with concentrated sulfuric acid.

3. REAGENTS

NOTE: DIW=deionized water

3.1 Stock phosphorus solution, 50 ppm: Dissolve .2197 g potassium dihydrogen phosphate (KH_2PO_4), dried at 105°C, in DIW and dilute to 1 liter.

3.2 Phosphorus mixed reagent: To 500 ml DIW add 70 ml conc. sulfuric acid (H_2SO_4)(3.6), .14 g of antimony potassium tartrate ($K(SbO)C_4H_4O_6 \cdot \frac{1}{2}H_2O$) and 6 g of ammonium molybdate ($(NH_4)_6Mo_7O_{24} \cdot 4H_2O$). Cool and dilute to 1 liter. (keep refrigerated)

3.3 Phosphorus color reagent: Dissolve .5 g of ascorbic acid in 100 ml of phosphorus mixed reagent (3.2). Prepare fresh the day of analysis.

3.4 Potassium persulfate: Use "Baker Instra-Analyzed" grade (for trace phosphate determinations).

3.5 Sodium hydroxide, 10 N: Dissolve 400 g NaOH in DIW. Cool and dilute to 1 liter.

3.6 Sulfuric acid, concentrated: Use reagent A.C.S. grade. "Ashland Reagent" is recommended due to it's low phosphorus content.

3.7 Phenolphthalein indicator solution: Dissolve 1 g of phenolphthalein in 100 ml of ethyl alcohol.

4. EQUIPMENT

4.1 150 ml beakers

4.2 hot plate

4.3 50 ml graduated cylinder

4.4 macro pipet

4.5 eppendorf pipets

4.6 50 ml short form nessler tubes

4.7 test tube rack

4.8 10 cm quartz spectrophotometer cells

4.9 A spectrophotometer suitable for measurements at 650 and 880 nm and capable of accommodating a 10 cm cell.

4.10 All glassware should be acid-washed and used only for the determination of phosphorus. The beakers (4.1) should be filled with a 3-5% sulfuric acid solution and heated on the hot plate for at least 1 hour. The cylinder (4.3), tubes (4.6) and cells (4.8) should be filled with a 5% sulfuric acid solution and stored over night. The beakers and tubes should be cleaned after each use. The cylinder and cells, periodically.

5. QUALITY CONTROL

5.1 Method blank (MB): One per twenty samples (5%) or one per run, whichever is more frequent. MB=DIW carried through whatever preparation the sample receives. (MB analysis code is TP for Total Phosphorus, OP for Orthophosphate, DOP for Dissolved Orthophosphate)

5.2 Digested Blank (DB): One per run. DB=DIW carried through whatever digestion the sample receives. The difference between a DB and a MB, is the DB is prepared in the same glassware every time. Record DB in log book.

5.3 Filter Blank (FB): One per run. FB=DIW carried through whatever filtering the sample receives. The difference between a FB and a MB, is the FB is prepared in the same glassware every time. Record FB in log book.

5.4 Matrix spike (MS): One per twenty samples (5%) or one per run, whichever is more frequent. Recommended spiking level is .10 mg/l for T-P and .050 mg/l for O-P and D-O-P.

5.5 Matrix spike duplicate (MSD): One per twenty samples (5%) or one per run, whichever is more frequent. Recommended spiking level is .10 mg/l for T-P and .050 mg/l for O-P and D-O-P. Occasionally a client will request a duplicate sample analysis instead of a MSD.

5.6 EPA reference material (EPA#): Analyze one per form and one per run, immediately after the instrument calibration. If the sample requires digestion, digest the EPA. Do not filter the EPA.

5.7 Complete records in the instrument log book.

6. METHODOLOGY

6.1 Sample Handling and Preservation

6.1.1 Phosphorus

6.1.1.1 Samples should be preserved with 2 ml of conc. sulfuric acid (H_2SO_4) per liter at the time of collection and kept at 4°C. Analyze within 28 days.

6.1.2 Orthophosphate

6.1.2.1 Samples should be preserved by refrigeration at 4°C. Analyze within 48 hours.

6.2 Interferences

6.2.1 No interference is caused by copper,iron,or silicate at concentrations many times greater than their reported concentration in sea water. However,high iron concentrations can cause precipitation of and subsequent loss of phosphorus. If a sample contains a high iron concentration, do not filter the sample before color development.

6.2.2 The salt error for samples ranging from 5 to 20% salt content is less than 1%.

6.2.3 Arsenate is determined similarly to phosphorus and should be considered when present in concentrations higher than phosphorus. However, at concentrations found in sea water, it does not interfere.

6.3 Sample Preparation

6.3.1 Phosphorus

6.3.1.1 Add .5 ml of conc. sulfuric acid (H_2SO_4)(3.6) to a 50 ml sample in a 150 ml beaker.

6.3.1.2 Add .4 g (one scope) of potassium persulfate (3.4).

6.3.1.3 Boil gently on a pre-heated hot plate until a final volume of about 10 ml is reached. Do not allow sample to go to dryness.

6.3.1.4 Cool and dilute the sample to about 20 ml. Add one drop of phenolphthalein indicator (3.7) and adjust to a pink color using 10 N NaOH (3.5). Dilute to 50 ml and mix.

6.3.1.5 Transfer a portion of the sample to a nessler tube (25 ml maximum), dilute to 50 ml and mix.

6.3.1.6 Determine phosphorus as outlined in 6.4

6.3.2 Hydrolyzable Phosphorus

6.3.2.1 Add .5 ml of conc. sulfuric acid (H_2SO_4)(3.6) to a 50 ml sample in a 150 ml beaker.

6.3.2.2 Boil gently on a pre-heated hot plate until a final volume of about 10 ml is reached. Do not allow sample to go to dryness.

6.3.2.3 Cool and dilute the sample to about 20 ml. Add one drop of phenolphthalein indicator (3.7) and adjust to a pink color using 10 N NaOH (3.5). Dilute to 50 ml and mix.

6.3.2.4 Transfer a portion of the sample to a nessler tube (25 ml maximum), dilute to 50 ml and mix.

6.3.2.5 Determine phosphorus as outlined in 6.4

6.3.3 Orthophosphate

6.3.3.1 Transfer 50 ml of sample to a nessler tube.

6.3.3.2 Determine phosphorus as outlined in 6.4

6.4 **Procedure**

6.4.1 Prepare a calibration blank and the following standards:

µl stock standard (3.1) diluted to 50 ml	concentration mg P/L
5	.005
10	.010
25	.025
50	.050
100	.10
250	.25

NOTE: Prepare either one 100 ml or two 50 ml calibration blanks, so as to have adequate volume to fill two 10 cm cells. This is needed to zero the instrument.

6.4.2 Add 8 ml of phosphorus color reagent (3.3) per 50 ml to the standards and samples, mix thoroughly. Allow 30-45 minutes for complete color development.

6.4.3 Measure the color absorbance of each standard and sample at 880 nm in a 10 cm cell, using the calibration blank as the reference solution.

6.5 **Calculation**

6.5.1 Using the standard concentrations and absorbances calculate a linear regression equation.

mg/l=(Abs.-constant)/X coefficient

Compute the sample concentrations using the above equation.

6.5.2 Lotus spreadsheets are available for doing these calculations. See T:\T-P.WK1, T:\O-P.WK1, or T:\D-O-P.WK1

7. **ANALYSIS TIME**

7.1 Since this method is performed manually, analysis time is dependent on run size and analyst speed.

8. **REFERENCES**

8.1 Methods for "Chemical Analysis of Water and Wastes", March 1979, EPA publication #600/4-79-020, Revised March 1983.

8.2 Standard Methods "For the Examination of Water and Wastewater", 16th Edition, copyright 1985, American Public Health Association, Washington, DC 20005.

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP No. **LTL-9109**

Previous SOP No. **LX-0041**

Title: **Ammonia Analysis by Lachat Auto-Analyzer, EPA Method 350.1**

Rev: 1

Laucks is in the process of re-numbering our SOPs. As an interim measure, this page serves as the cover page for those SOPs whose header information has not been updated. This page details the title, the SOP number that it is being controlled under, and the previous SOP number. The previous SOP cover sheet has been manually corrected to reflect the change but each page header will reflect the old numbering system. As SOPs are revised, the full header and cover page will be updated.

UNCONTROLLED

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #: ~~LX-0041~~ 12/26/95
LTL-9109

Title: Ammonia Analysis by Lachat Auto-Analyzer, EPA Method 350.1

Revision history:

Number	Date
1.0	3/30/94

Written by: Michelle L. L. Walberg — Date: 3/30/94

Reviewed by: Bill Lundberg, Inorganic Chemistry Supervisor — Date: 3/30/94

Reviewed by: Harry Romberg, QC Officer — Date: 3/30/94

Reviewed by: Mike Nelson, Technical Director — Date: 3/30/94

Approved by: John Buerger, Laboratory Director — Date: 3/30/94

1. Introduction and Scope

1.1. This method is for the measurement of ammonia as nitrogen in drinking, surface, and saline waters, domestic and industrial wastes in the range of 0.01 to 2.0 mg/L NH_3 as N by EPA Method 350.1. Higher concentrations can be determined by sample dilution. Soil leachates and distillates may also be analyzed by this method.

1.2. This analysis is based on the Berthelot reaction. Ammonia reacts with alkaline phenol, then with sodium hypochlorite to form indophenol blue. Sodium nitroprusside (nitroferricyanide) is added to enhance sensitivity. The absorbance of the reaction product is measured at 630 nm, and is directly proportional to the original ammonia concentration.

1.3. Interferences

1.3.1. Calcium and magnesium ions may be present in concentration sufficient to cause precipitation problems during analysis. The 5% EDTA solution is used to prevent the precipitation of calcium and magnesium ions from river water and industrial waste.

1.3.2. Sample turbidity may interfere with this method. Turbidity must be removed by filtration through a 0.45 µm filter prior to analysis.

1.3.3. Sample color that absorbs in the photometric range used will also interfere.

2. SAFETY

2.1. Care should be taken when working laboratory reagents and acids.

2.2. Wear a lab coat and safety glasses while in the lab and especially during any type of analysis.

2.3. Gloves offer protection from reagents while degassing and handling samples.

2.4. If it is necessary to open the back of the instrument, disconnect the power first.

3. REAGENTS

3.1. Ammonia-free de-ionized water.

3.2. Sodium phenolate - Dissolve 83.0g crystalline phenol and 32.0g sodium hydroxide in DIW. Dilute to 1.0L and mix until dissolved.

3.3. Sodium hypochlorite - Dilute 500 mL of 5.25% NaOCl (bleach) to 1.0 L with DIW.

3.4. Buffer - Dissolve 50.0g of EDTA and 5.5g of NaOH with DIW. Dilute to 1.0 L with DIW and mix until dissolved (about one hour).

3.5. Sodium nitroferricyanide - Dissolve 3.50 g of sodium nitroferricyanide in DIW. Dilute to 1.0 L. Make this reagent before it is needed. Greater sensitivity is achieved with 2-day old reagent.

3.6. Carrier (0.2% H_2SO_4) - Dilute 3.0 mL H_2SO_4 to 1.5 L with DIW.

3.7. Stock NH_3 solution - Dissolve 0.3819g of anhydrous ammonium chloride, NH_4Cl, dried at 105 °C, in DIW, and dilute to 1.0 L. 1.0 mL of stock standard equals 0.1 mg NH_3-N (100 ppm solution.).

3.8. concentrated H_2SO_4 (for soil distillation)

3.9. Phenolphthalein indicator (for soils)

3.10. Sodium borate buffer (for soils) - Dissolve 9.5 g sodium borate in DIW, add 88 mL 0.1N NaOH, and dilute to 1.0L. Keep in an amber glass bottle.

3.11. Anti-foam solution, prepared (for soils)

3.12. 50 % NaOH (for soils)

3.13. 10,000 ppm NH_3 standard for spiking soils

4. EQUIPMENT

4.1. Lachat Automatic Flow Injection Ion Analyzer

4.2. Automatic sampler

4.3. Peristaltic pump

4.4. Injection module: 150 cm, 0.81 mm i.d., sample loop

4.5. Colorimeter: 10 mm flow cell, 630 nm filter

4.6. Heating block: 60 °C

4.7. Reaction manifold 10-107-06-1-B or equivalent

4.8. Sample and calibration racks, with calibration vials

4.9. Helium tank for degassing reagents

4.10. Fritted gas dispersion tube

4.11. PC with keyboard and printer (IBM compatible)

4.12. Various micropipettes with tips

4.13. 13 x 100 borosilicate disposal test tubes

4.14. 100 mL volumetric flask

4.15. Kjeldahl flasks (for soil distillations)

4.16. 250 mL beakers (for soils)

5. QUALITY CONTROL

5.1. One initial calibration verification (ICV)sample must be analyzed at the beginning of each analytical run, and the value must fall within set limits.

5.2. An initial calibration blank (ICB) must follow the ICV, and must be less than twice the LLD (less than 0.02 mg/L).

5.3. A method detection limit (MDL)sample must be analyzed with each analytical run. The minimum requirement for the MDL is that it be detectable, although it is desirable that it be within the range of 64% to 220% of the true value (0.01 mg/L).

5.4. A continuing calibration verification (CCV) sample must be analyzed at a frequency of 10% and at the end of the run, and must be +/- 15% of the true value. A suggested value would be 0.5 mg/L.

5.5. A continuing calibration blank (CCB) must follow each CCV, and must be less than twice the LLD.

5.6. Matrix spikes and their duplicates (MS/MSD) must be analyzed at a frequency of 5%, and recovery limits and RPD values must fall within set limits.

6. PROCEDURE

6.1. Sample Handling

6.1.1. Preserve sample by the addition of 2.0 mL conc. H_2SO_4 per liter.

6.1.2. Refrigerate samples at 4 °C.

6.2. Instrument set-up

6.2.1. Turn on power and all modules, and allow heating block to warm up to 60 °C

6.2.2. Degas all reagents.

6.2.3. Place reagent feed lines into proper containers, and raise tension levers on pump tube cassettes.

6.2.4. Allow reagents to pump through system until a stable baseline in obtained.

6.3. Calibration

6.3.1. Dilute 2 mL of the 100 ppm stock NH_3 standard up to 100 mL with DIW in a volumetric flask. This is your 2 ppm working standard.

6.3.2. From the above stock solution, make the following calibration standards:

2.0 ppm	undiluted
0.50 ppm	5.0mL diluted to 20mL with carrier

0.10 ppm	1.0mL diluted to 20mL with carrier
0.05 ppm	0.5mL diluted to 20mL with carrier
0.00 ppm	blank, straight carrier

6.3.3. Place calibration standards on autosampler, and start calibration.

6.4. Soil preparation

6.4.1. Though soil samples can be prepared by either distillation or by leaching them with 0.2% H_2SO_4, distillation is the preferred method, and is what is discussed here.

6.4.2. Into a Kjeldahl flask place 2.0g of sample, 300 mL of DIW, some boiling stones, 2 drops of phenolphthalein indicator, 25 mL of sodium borate solution, and a squirt of anti-foam solution.

6.4.3. Samples should be pink. If they are not, add 50% NaOH drop wise until color forms.

6.4.4. Also distill a preparation blank, a matrix spike sample, and it's duplicate.

6.4.5. Spike MS/MSD with 0.1 mL of the 10,000 ppm solution, for an NH_3 spiking value of 5 ppm at the instrument.

6.4.6. Place 50 mL of 0.8% H_2SO_4 into 250 mL beakers.

6.4.7. Distill flasks into beakers until distillate levels reach approximately. 175-195 mL.

6.4.8. Turn off heat and pull beakers down so as not to draw distillate up the condenser.

6.4.9. Dilute distillates to 200 mL each, and analyze the same as waters.

6.5. Sample analysis

6.5.1. Place all prepared samples in test tubes and on the autosampler, and start tray analysis.

6.5.2. Dilute samples as needed, and re-analyze.

6.5.3. All need calculations to reach a final mg/L value are done by the computer program.

6.5.4. For soil results, take the mg/L value from the analysis, multiply by the final distillate volume (usually 200 mL), divide by the initial sample weight (usually 2.0g), and divide by the sample's total percent solids value.

7. ANALYSIS TIME

7.1. Barring any problems or excessive dilutions, approximately 20 samples with their corresponding QC can be analyzed in 90 minutes.

8. REFERENCES

8.1. Lachat QuikChem Method No. 10-107-06-1-B

8.2. EPA Method 350.1

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-9125

EPA 353.2 Nitrate/Nitrite as N by Lachat

Revision history:

Number	Date
0	04/23/96
1	10/28/98

UNCONTROLLED

Written by: Bill Lundberg Date: 10/27/98
Bill Lundberg, Inorg Div. Manager

Approved by: Harry Romberg Date: 10-27-98
Harry Romberg, QC Officer

Approved by: Kathy E Kreps Date: 10/28/98
Kathy Kreps, Laboratory Director

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1. Introduction and Scope

1.1. Method Description

1.1.1. This method is applicable to the determination of nitrate/nitrite in drinking, surface and saline waters, domestic and industrial wastes. The method is applicable in the range from 0.010 to 2.00 mg/L as N. Higher nitrate/nitrite concentrations can be determined by diluting the sample. The nitrate in the sample is quantitatively reduced to nitrite by passing it over a cadmium reduction column. The diazonium compound formed by diazotation of sulfanilamide by nitrite in water under acid conditions is coupled with N-(1-napthyl)-ethylenediamine dihydrochloride to produce a reddish-purple color which is measured in a spectrophotometer at 520 nm.

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 analysis.

1.2. Sample Collection, Sample Storage, Holding Times

1.2.1. Samples are collected in plastic 500 mL bottles. They are preserved with H_2SO_4 to pH <2. The samples should be held at 4±2 C° until analysis. The holding time is 28 days, based on time of collection.

1.3. Definition of Terms

1.3.1. This section defines terms and acronyms as they are used in this SOP. Other terms, such as MS/MSD or method blank, are not defined here since it is assumed that the user of this SOP already understands their more general meaning.

1.3.2. **Batch Identifier** - A number given to each preparation or analysis group which uniquely identifies that batch. This number is an analysis number which is similar to the blank ID, only preceded by an "A" rather than a "B" for inorganic batches. The preparation batch IDs

are discussed in other documentation. The batch identifier for the third batch of waters run on Feb 29,1996 would be: A022996LANNW03

1.3.3. **Blank spike** - A background free matrix (DIW for water, clean sand for soils/sediments) to which known amounts of target analytes are added each time samples are prepared. Blank spikes are required on all HAZWRAP and NFESC work. Note that an LCS or SRM (see below) will substitute as a blank spike for most inorganic analyses. In the context of this SOP, a blank spike is the same as a QC check standard. See also QC check standard.

1.3.4. **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.

1.3.5. **CCV** - Continuing calibration verification. - This is the same acronym used in the CLP program. This is a standard analyzed at some prescribed frequency (immediately following calibration, 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.

1.3.6. **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.

1.3.7. **Corr Coef, CC** - Correlation coefficient - A measure of the "goodness of fit" of a set of data to a regression model. The closer the value is to 1, the higher the degree of confidence in the correlation

1.3.8. **DIW** - Deionized water - Lab reagent water. This water should be free of virtually all analytes.

1.3.9. **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.

1.3.10. **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. For most inorganic methods, this verification standard is from a source different from that used to make the calibration standards.

1.3.11. **IDL** - Instrument detection limit. The lowest concentration of a target analyte that will yield a signal:noise ratio of least 3x. Used as a starting point for selecting MDL study spiking levels.

1.3.12. **MDL** - Method detection limit - The lowest concentration a sample which will yield a positive result that is greater zero at a known level of confidence. MDLs are empirically determined by Laucks.

1.3.13. **MDL standard** - Method detection limit standard - A standard prepared so that the concentrations of the target analytes are no greater than 4x the empirically determined MDLs. This standard is used to verify that the instrument or system is capable of detecting the target analytes on an ongoing basis.

1.3.14. **QC check standard** - Quality control check standard. Referred to in this SOP as a blank spike. A QC check standard is used to determine whether the analytical system is in control if MS/MSD recoveries are out of control. See also blank spike.

1.3.15. **SRM or LCS** - Standard Reference Material or Laboratory Control Sample. This is a material of approximately the same matrix as the samples, containing a known and usually certified amount of target analyte and which is prepared and analyzed in the same manner as a typical sample. This sample is used to demonstrate that the analytical system is in control

1.3.16. **QC period** - Quality control period - An analysis sequence initiated by the analysis of one or more standards, followed by samples, and terminated with a standard and blank analysis. A QC period can be open-ended chronologically, but calibration verification must be documented using the procedures in this SOP

1.3.17. **RSD or %RSD** - Relative standard deviation or percent relative standard deviation - The ratio of the standard deviation of a set of values to the mean of the set of values. A measure of the similarity of the values one to another.

1.3.18. **Sequence** - A set of sample extracts and standard solutions injected into an instrument in a chronologically continuous group. See also QC period.

2. Equipment List and Standards

2.1. Instrument

2.1.1 Lachat QuickChem AE-200

2.1.2 Reaction Module 10-107-04-1-C

2.1.3 Cd/Cu column. This is purchased pre-packed from Lachat.

2.1.4 Autosampler

2.1.5 Proportioning Pump

2.1.6 IBM PC with software for capturing and reducing the data.

2.2. Standards

2.2.1. Stock Nitrate solution: A commercially prepared solution of 100 mg/L NO_3-N is purchased annually. This solution will be preserved with 0.2% $CHCl_3$

2.2.2 Intermediate Nitrate Standard: Dilute 2000 µLs of 2.2.1 to 100 mLs in a volumetric flask with 0.2 % H_2SO_4. The concentration of this solution is 2.0 mg/L. This solution should be prepared weekly.

2.2.3 Working standards: Dilute 0, 500µl, 1000µl, 5.0 mL, and 20.0 mL of 2.2.2 to 20 mLs with 0.2% H_2SO_4. The concentration of these solution are zero, .050 mg/L, .100 mg/L, .500 mg/L and 2.000 mg/L. These solutions should be made fresh daily. The CCV solution is the .500 mg/L standard. MS/MSD samples are prepared by diluting 25µl of 2.2.1 to 5.0 mLs with the sample. The MDL solution is prepared by diluting 25 µl of 2.2.2 into 5.0 mLs of 0.2 % H_2SO_4.

2.3 Reagents

2.3.1 Ammonium chloride buffer: Dissolve 85. g of NH_4Cl and 1.0 g EDTA in approx 900 mLs DIW. Adjust the pH to 8.5 with 15M NaOH. Dilute to 1L and mix.

2.3.2 Sulfanilamide color reagent: In a 1L volumetric flask, combine 600 mL of DIW, 100 mL of 85% H_3PO_4, 40.0 g of sulfanilamide, and 1.0 g N-1-napthylethylenediamine dihydrochloride. Stir for 20 mins, or until dissolved. Dilute to 1L and mix.

2.3.3 Carrier: Dilute 3 mL of conc. H_2SO_4 to 1.5 L with DIW.

3. Safety precautions and Waste Disposal

3.1. Safety Precautions

3.1.1. All standards, samples and sample solutions should be handled as if they are hazardous substances.

3.1.2. Refer to the instrument manufacturer's manual for routine instrument precautions.

3.1.3. Routine precautions include an awareness of the moving parts on the instrument you're using. These parts are often charged with power from an electrical component or with high pressure gas and have the potential to do harm if not used properly.

3.1.4. Electrical shock - All instruments present the possibility of electrical shock The operator should take all precautions including ensuring that all instruments are operated with fully grounded power outlets, turning off the instrument and disconnecting the instrument from the electrical power supply before working on any electrical components, etc.

3.1.5 Sodium hydroxide and sulfuric acid are extreme irritants if they come in contact with skin or eyes. When using these chemicals, the analyst should wear gloves and safety glasses.

3.2. Waste Disposal

3.2.1. Waste from the instrument is carried by a drain to the sink. Leftover sample aliquots are also disposed of into the sink..

3.2.2. Waste segregation and disposal from the point of collection is further covered in the Laucks SOP on Waste Segregation and Disposal.

4. Calibration and Quality Control

4.1. *Method Detection Limit Study*

4.1.1. Prior to the analysis of any samples, it is necessary to establish method detection limits. This procedure is fully described in Laucks 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.

4.2. *Initial Multi-Point Calibration*

4.2.1. Analyze standard solutions using at least 4 different concentration levels. The calibration curve was described in the Standards section. The lowest standard is at .050 mg/L. The highest standard is 2.00 mg/L. The calibration solutions are analyzed from the highest concentration to the lowest

4.2.2. Criteria

4.2.3. Initial calibration data is evaluated using the correlation coefficient of a linear regression analysis. The correlation coefficient must be 0.995 or greater for a 5-point calibration. All CCVs and sample concentrations must be computed using the regression equation.

4.2.4. Corrective action

4.2.5. If the criteria are not met, the instrument must be recalibrated.

4.3. *Initial Calibration Verification*

4.3.1. Immediately after the calibration curve, analyze a standard from a source other than that from which the calibration material was obtained.

4.3.2. Criteria

4.3.3. The calculated concentration of the ICV must be within the limits supplied by the manufacturer or should not exceed 90%-110% of the true value if no limits are provided.

4.3.4. Corrective action

4.3.5. If the ICV criteria are not met, no samples can be analyzed. Perform system maintenance and re-check the ICV. If the criteria still cannot be met, the system must be recalibrated.

4.4. *Initial Calibration Blank*

4.4.1. After the analysis of the ICV standard an instrument blank (ICB) is analyzed. The absolute value of NO_2/NO_3 in the CCB should not exceed the reporting limit.

4.4.2. Corrective action

4.4.2. If the initial CCB contains target analyte levels above the reporting limit, the system is out of control. The source of contamination must be identified and corrected before proceeding with the analysis.

4.5. Method Detection Limit Standard

4.5.1. After the analysis of the ICV and the ICB, but before the analysis of any samples, an MDL standard is to be analyzed. The MDL standard is used to provide on-going verification of the ability of the system to detect analytes at a concentration near the method detection limit. The MDL standard should give a positive result. It must be detected for the system to be considered in control.

4.5.2. Corrective Action

4.5.3. If NO_2/NO_3 is not detected, the analysis must be terminated until the problem has been solved. Alternatively, if the affected samples are well above the detection limit (i.e. bracketed by appropriate standards), they may be reported. No undetected values should be reported if the MDL standard for that analyte(s) is undetected.

4.6. *Continuing Calibration Verification (CCV) and Blank (CCB)*

4.6.1. A mid-range calibration standard is analyzed after calibration and after every 10 samples. Immediately following the CCV, a blank solution is analyzed. In addition, this standard and blank must be the last samples analyzed in the run.

4.6.2. The CCV must fall within ± 10% of the true value.

4.6.3. The levels of NO_2/NO_3 in the CCB should not exceed the reporting limit.

4.6.4. Corrective action

4.6.5. 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.

4.6.6. If the initial CCB contains target analyte levels above the detection 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 CCBs which are within limits.

4.7. *Method Blanks*

4.7.1. Method blanks are used to verify contamination free reagents and apparatus. They are prepared with every set of samples prepared at the same time or at least one blank every 20 samples which ever is more frequent. Any analyte response above the detection limit is reported. Method blank control limits are that contamination should not exceed the reporting limit

4.7.2. Corrective action

4.7.3 .Corrective action may necessitate re-preparation and re-analysis of the sample set. For example if an analyte were found in the blank but not in any of the associated samples then sample group may not require re-analysis. In addition, if sample levels exceed 20 times the blank, the level of contamination may be considered insignificant. In any case, if re-preparation and re-analysis is not being undertaken, the analyst must first discuss the issue with the Quality Control Officer. It is the laboratory's responsibility to ensure that method interferences caused by contaminants in acids, solvents, reagents, glassware, and other sample processing hardware leading to discrete artifacts and/or elevated baselines in the analytical run be minimized. In the extreme case of chronic contamination, blanks may have to be analyzed from each stage of the sample processing to determine the contamination source so it can be eliminated. In all cases where blank contamination exceeds the control limit, a narrative comment must be made which documents the corrective actions taken.

4.8. *Matrix Spike*

4.8.1. 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 analysis such that the final solution is fortified at 0.500 mg/L. The analyst should attempt to avoid selecting samples which are identified by the client as blanks. As the purpose of the matrix spike is to test the system under "typical" conditions, the analyst may also avoid selecting the most difficult sample of the batch for spiking. It is always required that a matrix spike analysis be performed with each preparation/analysis batch, however, the minimum frequency for MS analysis is 1 each per 20 samples per matrix.. This matrix spike sample is used to evaluate the matrix effect of the sample upon recovery of the analytes. The recovery of spike analytes is calculated as follows:

$$\% \text{ recovery} = \frac{(SS - S) * 100}{SA}$$

where:

SS = concentration in spiked sample
S = native concentration in unspiked sample
SA = spiked added, the amount of spiking material actually added to the sample calculated on the sample basis

4.8.2. The recovery criteria are detailed in the current Control Limits Catalog and in the Quality Control Database and will change from time to time.

4.8.3. Corrective action

4.8.4. 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 the matrix spiking compounds from the SRM and/or blank spike analysis. If the SRM is in control, the out of control recovery is due to matrix interference and probably does not warrant reanalysis. In all cases a narrative explanation of the condition is required to detail the corrective actions taken.

4.9. *Matrix Spike Duplicate*

4.9.1. The compound recovery criteria are identical to those for the matrix spike sample. In addition, the matrix spike duplicate is used measure method precision. This is done by computing the relative percent difference (RPD) between the matrix spike and matrix spike duplicate recovery values. MS/MSD is the preferred method for measuring precision, however if the client requests, or if it is to reported in CLP format, MS/DUP is performed. This calculation is as follows:

$$RPD = \frac{|S1 - S2| * 100}{(S1 + S2)/2}$$

where:

S1 = measured concentration for MS sample
S2 = measured concentration for MSD sample

4.9.2. RPD control limits are detailed in the current Control Limits Catalog and in the Quality Control Database (QC_DB) and will change from time to time.

4.10. *Sample Duplicate*

4.10.1. Criteria

4.10.2. Sample duplicates are required only when the client requests, when CLP practices are employed, or when the method specifically calls for duplicates. At least one duplicate sample per 20 samples per matrix is required when matrix spikes/duplicates are being performed. RPD values are calculated in a manner similar to MS/MSD RPDs:

$$RPD = \frac{|S1 - S2| * 100}{(S1 + S2)/2}$$

where:
S1 = measured concentration in the initial analysis
S2 = measured concentration in the duplicate analysis

4.10.3. The RPD control limits are detailed in the current Control Limits Catalog and in the Quality Control Database and will change from time to time.

4.10.4. Corrective action

4.10.5. If a trend in out of control RPD values is observed, the methods used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable reproducibility. Generally, if recoveries are in control and no analyte of interest was detected in any of the samples, no immediate action will be taken on that sample set. If integrity of reported sample values is in doubt, re-analysis may be called for. Corrective actions should be discussed with the Quality Control Officer.

5. Operation procedures

5.1. Sample Analysis

5.1.1. Analysis sequence

S0
S1
S2
S3
S4
ICV
CCV0
ICB
MDL
PB
Sample A
Sample A Spike
Sample A Spike Dup (or Dup)
Sample B
etc.
after 10 samples
CCV1
CCB1

A CCV and CCB must be the last samples analyzed..

5.2. Instrumental Conditions

5.2.1. Proportioning pump set at 35.

5.2.2 Injection valve with 17 cm of 0.8 mm i.d. tubing.

5.2.3 Reaction Module 10-107-04-1-C

5.2.4 Colorimeter with 520 nm filter, and 10 mm flow cell.

5.3 Analytical Operation

5.3.1 Turn on the system at the power strip, and allow to warm up for 5-10 mins.

5.3.2 Download the method by selecting "NN" in the Analysis Select and Download screen.

5.3.3 Place reagent feed tubes into the proper containers.

5.3.4 Lock pump tube cassettes down on the pump, and raise the tension levers to vertical.

5.3.5 Pump reagents until all air is expelled from the tubing.

5.3.6 Stop the pump, place the cadmium column in line on the manifold, and restart the pump.

5.3.7 Make up the standards as described in the standards section.

5.3.8 Begin calibration by selecting " Calibrate Now" under the "Submit" menu. Follow instructions on the screen to begin calibration.

5.3.9 After calibration approval, verify that the correlation coefficient is >0.995.

5.3.10 Load samples into a tray and record their position in the "Identification" field of the "Submit" menu Record any dilutions made in the "Dilution" field.

5.3.11 Submit the tray by choosing "Submit Current Tray". Give the appropriate responses to the prompted fields.

5.3.12 After analysis is complete, move to the "Results/Approval" field. To print the report, choose Tray selection, Sample tray, Reports, Print reports.

5.3.13 After analysis is completed, stop pump, remove the Cadmium column, place the reagent tubes in DIW and pump for 5 mins to clean the system.

5.3.14 Remove the tubes from the DIW, pump air until all liquid is expelled, unlatch the pump tubes, and turn off the power strip.

5.4 Compound Quantification

5.4.1 The instrument report is in mg/L. This value is corrected for any dilution inputted into the software. Any external dilutions need to be corrected for on the report. Soil samples are reported in mg/Kg DB using the following equation:

$$\text{Nitrate/Nitrite, mg/Kg DB} = \frac{\text{N/N in extract* dilution}}{\text{\% TS/100}}$$

5.4.2 Any samples having concentrations above the high standard are diluted with 0.2% H_2SO_4 and reanalyzed.

6. Reports

6.1. Data Packet Organization

6.1.1. A data package will consist of a copy of the calibration, a copy of the quantitation report for the samples, a data summary showing any external calculations (i.e., TS corrections), and a QC summary report showing where the records are located in the QC data base. A CLP like package will also require a runlog showing all the standards preparation, standard and QC solution references, and the analytical sequence and it's timing.

6.2. Quality Control Reports

6.2.1. All results for quality control tests are entered into the lab data base. A report of all data entered must be included in the data packet. The routine minimum is a method blank report, and an MS/MSD or MS/duplicate report. Many analyses will also require an SRM, blank spike or other report.

6.3. Sample Result Reports

6.3.1. Data Qualifying Flags

6.3.1.1. Sample report results are qualified with data qualifying flags. These flags have the following definitions:

CODE	Definition
U	: The analyte of interest was not detected, to the limit of detection indicated.

6.4. Control Chart(s)

6.4.1. The recovery values for nitrate/nitrite in the SRM are plotted on control charts.

Appendix I

Laucks Testing Laboratories

Method 353.2 NO3/NO2

QA Requirements and Corrective Actions

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Initial Calibration	3 pt calibration	4 pt calibration R≥0.995	Daily	Recalibrate	Copy of calibration in package
Initial Calibration Verification	90-110%	90-110% or manufactures limits	1/20	Recalibrate	Included in raw data
Initial Calibration Blank	< MDL	Less than the reporting limit	Once per run	Reanalyze or recalibrate	Included in raw data
Continuing Calibration Verification	90-110%	90-110 %	Every 10 samples, after cal., and at the end	Recalibrate	Included in raw data
Continuing Calibration Blank	<MDL	Less than the reporting limit	Every 10 samples and at the end	Recalibrate	Included in raw data
MDL standard recovery	None required	Must be detectable	Once per run	Recalibrate	Included in raw data
Method Blank	1/ batch < MDL	Less than the reporting limit	One per 20	Reanalyze or recalibrate unless sample>20X MB	Included in raw data
Matrix Spike	10% frequency 90-110%	See QC database	* One per 20	Reanalyze	QC database
Matrix Spike Duplicate	None required	See QC database	* One per 20	Reanalyze	QC database

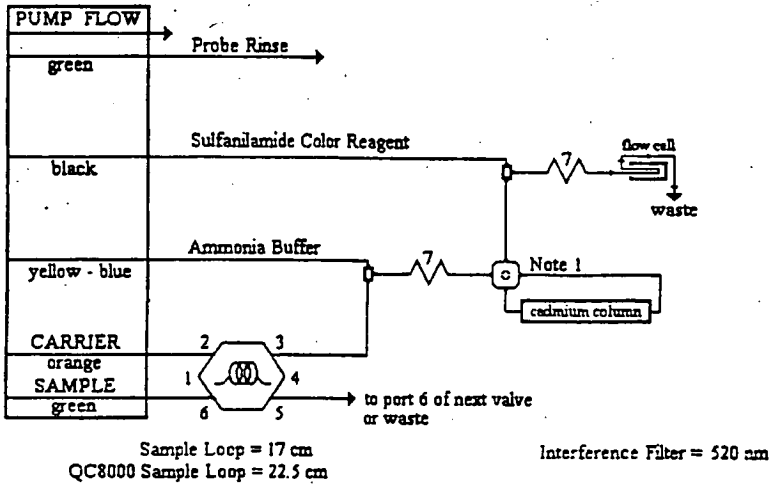
Duplicate % Difference	None required	See QC database	* One per 20	Reanalyze	QC database
Lab Fortified Blank	1/ batch 90-110%	This is the same as the ICV			

* Either a MS/MSD or a MS/DUP will be run with every batch

Appendix II

Manifold Schematic from Current Instrument Manual

NITRATE MANIFOLD DIAGRAM



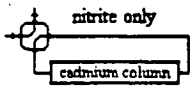
CARRIER is DI Water.

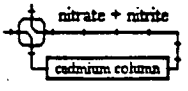
All manifold tubing is 0.8 mm (0.032 in) i.d. This is 5.2 uL/cm.

7 is 135 cm of tubing on a 7 cm coil support

APPARATUS: An injection valve, a 10 mm path length flow cell , and a colorimetric detector module is required.

Note 1: This is a 2 state switching valve used to place the cadmium column in-line with the manifold.





LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-9133

Title: **DETERMINATION OF TOTAL KJELDAHL NITROGEN IN WATER BY EPA 351.2**

Revision history:

Number	Date
0	12/14/98

Written by: Lila K Hepburn — Date: 12/15/98
Lila Hepburn, Chemist

Reviewed by: Bill Lundberg — Date: 12/15/98
Bill Lundberg, Inorganic Supervisor

Approved by: Harry Romberg — Date: 12-15-98
Harry Romberg, QA Officer

Approved by: Kathy Kreps — Date: 12-15-98
Kathy Kreps, Laboratory Director

UNCONTROLLED

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APPENDIX I **15**

1. Introduction and Scope

1.1 Method Description

1.1.1 This method covers the determination of total Kjeldahl nitrogen in drinking, ground, and surface waters, domestic and industrial wastes. The procedure converts nitrogen components of biological origin such as amino acids, proteins, and peptides to ammonia, but may not convert the nitrogenous compounds of some industrial wastes such as amines, nitro compounds, hydrazones, oximes, semicarbazones and some refractory tertiary amines. The applicable range is 0.1 to 20 mg/L TKN. The range may be extended by sample dilution.

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 analysis.

1.2 Sample Collection, Sample Storage, Holding Times

1.2.1 Samples should be collected in plastic or glass bottles. They are preserved with H_2SO_4 to pH <2 and cooled to 4 ± 2° C at time of collection. Samples are kept at 4 ± 2° C until analysis. Holding time is 28 days based on time of collection.

1.3 Definition of Terms

1.3.1 This section defines terms and acronyms as they are used in this SOP. Other terms, such as MS/MSD or method blank, are not defined here since it is assumed that the user of this SOP already understands their more general meaning.

1.3.2 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 an analysis number which is similar to the blank ID, only preceeded by an "A" rather than a "B" for inorganic batches. The preparation batch IDs are discussed in other documentation. The batch ID for the second set of water samples analyzed on 12/14/98 would be A121498AEW02

1.3.3 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.

1.3.4 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.

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1.3.9 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. For most inorganic methods, this verification standard is from a source different from that used to make the calibration standards.

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1.3.13 QC period - Quality control period - An analysis sequence initiated by the analysis of one or more standards, followed by samples, and terminated with a standard and blank analysis. A QC period can be open-ended chronologically, but calibration verification must be documented using the procedures in this SOP

1.3.14 RSD or %RSD - Relative standard deviation or percent relative standard deviation - The ratio of the standard deviation of a set of values to the mean of the set of values. A measure of the similarity of the values one to another.

1.3.15 Sequence - A set of sample extracts and standard solutions injected into an instrument in a chronologically continuous group. See also QC period.

2. Equipment List and Standards

2.1 Instrument

2.1.1 Lachat QuickChem AE-200

2.1.2 Reaction Module 10-107-06-2-D

2.1.3 Proportioning pump

2.1.4 IBM PC with software for capturing and reducing the data

2.1.5 BD-46 block digestor

2.2 Standards

2.2.1 Stock NH_3-N solution: Dissolve 0.3819g of NH_4Cl and 2.0 mL of H_2SO_4 in DIW and dilute to 1L. The concentration of this solution is 100. mg/L. This solution is prepared annually.

2.2.2 Intermediate TKN standard: Dilute 20.0 mL of 2.2.1 and 30.0 mL digestion solution to 100 mL with DIW. This solution has a concentration of 20.0 mg/L and should be prepared weekly.

2.2.3 Working standards: Dilute 0, 0.100 mL, 0.500 mL, 1.000 mL, 5.0 mL, and 20.0 mL of 2.2.2 to 20.0 mL with TKN diluent. The concentration of these standards are zero, 0.100 mg/L, 0.500 mg/L, 1.00 mg/L, 5.00 mg/L, and 20.0 mg/L. These solutions should be made fresh daily.

2.3 Reagents

2.3.1 Digestion Solution: Dissolve 268 g K_2SO_4, 14.6 g anhydrous $CuSO_4$, and 268 mL H_2SO_4 in DIW and dilute to 2 L.

2.3.2 Buffer: Dissolve 35. g $Na_2HPO_4 \bullet 7H_2O$ in DIW. Add 50. g KNa Tartrate and 50. g NaOH. When all reagents are dissolved dilute to 1L with DIW.

2.3.3 TKN Diluent: Dilute 375 mL of 2.3.1 to 1L.

2.3.4 0.8 N NaOH: Dissolve 32.0 g NaOH in 1L DIW.

2.3.5 Sodium Salicylate Solution: Dissolve 150. g sodium salicylate in ≈ 500 mls DIW. Add 1.00 g sodium nitroprusside. When reagents have dissolved, dilute solution to 1 L.

2.3.6 Hypochlorite: Dilute approximately 15 mL of Clorox bleach to 250 mL with DIW. Prepare daily. Do not degas.

Unless otherwise noted all reagents should be degassed for two minutes with He.

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 This procedure requires the use of a block digestor which heats to 380 F. Caution should be used when working with and around the block. Sample tubes are also very hot when removed from the digestor.

3.1.6 Concentrated sulfuric acid can cause severe burns. When working with it, gloves and safety glasses should be worn. Note: When samples are removed from the digestor, they contain mostly hot sulfuric acid.

3.2 Waste Disposal

3.2.1 Waste from the instrument is carried by a drain to the sink. Leftover sample aliquots are disposed of in the sink.

3.2.2 Waste segregation and disposal from the point of collection is further covered in the Laucks SOP on Waste Segregation and Disposal.

4. Calibration and Quality Control

4.1 Method Detection Limit Study

4.1.1 Prior to the analysis of any samples, it is necessary to establish method detection limits. This procedure is fully described in Laucks SOP on MDL studies. Briefly, it involves the analysis of 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 Initial Multi-Point Calibration

4.2.1 Analyze standard solutions using at least 5 different concentration levels. The calibration solutions are described in 2.2.3. Analyze the standard solutions from the highest concentration to the lowest.

4.2.2 Criteria

4.2.2.1 Initial calibration data is evaluated using the correlation coefficient of a linear regression analysis. The correlation coefficient must be 0.995 or greater for a 5-point calibration. All CCVs and sample extract concentrations must be computed using the regression equation.

4.2.3 Corrective action

4.2.3.1 If the criteria are not met, the instrument must be recalibrated.

4.3 Initial Calibration Verification

4.3.1 Immediately after the calibration curve, analyze a standard from a source other than that from which the calibration material was obtained.

4.3.2 Criteria

4.3.2.1 The calculated concentration of the ICV must be within the limits supplied by the manufacturer or should not exceed 90%-110% if no limits are provided.

4.3.3 Corrective action

4.3.3.1 If the ICV criteria are not met, no samples can be analyzed. Perform system maintenance and re-check the ICV. If the criteria still cannot be met, the system must be recalibrated.

4.4 Initial Calibration Blank

4.4.1 After the analysis of the ICV standard an instrument blank (ICB) is analyzed. The levels of target analytes in the CCB should not exceed the reporting limit.

4.4.2 Corrective Action

4.4.2.1 If the initial CCB contains target analyte levels above the reporting limit, the system is out of control. The source of contamination must be identified and corrected before proceeding with the analysis.

4.5 Continuing Calibration Verification (CCV) and Blank (CCB)

4.5.1 A mid-range calibration standard is analyzed after every 10 samples. Immediately following the CCV, a blank solution is analyzed. In addition, this standard and blank must be the last samples analyzed in the run.

4.5.1.1 The CCV must fall within ± 10% of the true value.

4.5.1.2 The levels of target analytes in the CCB should not exceed the reporting limit.

4.5.2 Corrective action

4.5.2.1 If CCV limits are exceeded, check calculations or perform instrument maintenance. Recalibrate and reanalyze. No sample results may be reported that are not bracketed by a successful calibration and a CCV which is in control or by preceding and following CCVs which are within limits.

4.5.2.2 If the initial CCB contains target analyte levels above 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 CCBs which are within limits.

4.6 Method Blanks

4.6.1 Method blanks are used to verify contamination free reagents and apparatus. They are prepared with every set of samples prepared at the same time or at least one blank every 20 samples which ever is more frequent. Any analyte response above the reporting limit is reported. Method blank control limits are that contamination should not exceed the reporting limit.

4.6.2 Corrective action

4.6.2.1 Corrective action may necessitate re-preparation and re-analysis of the sample set. For example if an analyte were found in the blank but not in any of the associated samples then sample group may not require re-analysis. In addition, if sample levels exceed 20 times the blank, the level of contamination may be considered insignificant. In any case, if re-preparation and re-analysis is not being undertaken, the analyst must first discuss the issue with the Quality Control Officer. It is the laboratory's responsibility to ensure that method interferences caused by contaminants in acids, solvents, reagents, glassware, and other sample processing hardware leading to discrete artifacts and/or elevated baselines in the analytical run be minimized. In the extreme case of chronic contamination, blanks may have to be analyzed from each stage of the sample processing to determine the contamination source so it can be eliminated. In all cases where blank contamination exceeds the control limit, a narrative comment must be made which documents the corrective actions taken.

4.7 Matrix Spike

4.7.1 A sample is chosen at random from the samples to be analyzed, and an aliquot of spiking solution is added to this sample prior to preparation. The analyst should attempt to avoid selecting 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. This matrix spike sample is used to evaluate the matrix effect of the sample upon recovery of the analytes. The recovery of spike analytes is calculated as follows:

$$\text{recovery} = \frac{(SS - S) * 100}{SA}$$

where:

SS = concentration in spiked sample
S = native concentration in unspiked sample
SA = spiked added, the amount of spiking material actually added to the sample calculated on the sample basis

4.7.2 The recovery criteria are detailed in the Quality Control Database (QC_DB) and will change from time to time.

4.7.3 Corrective action

4.7.3.1 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.

4.8 Matrix Spike Duplicate

4.8.1 The compound recovery criteria are identical to those for the matrix spike sample. In addition, the matrix spike duplicate is used measure method precision. This is done by computing the relative percent difference (RPD) between the matrix spike and matrix spike duplicate recovery values. This calculation is as follows:

$$RPD = \frac{|S1 - S2| * 100}{(S1 + S2)/2}$$

where:
S1 = measured concentration for MS sample
S2 = measured concentration for MSD sample

RPD control limits are detailed in the Quality Control Database (QC_DB) and will change from time to time.

5. Operation procedures

5.1 Sample Analysis

5.1.1 Analysis sequence

20.0 mg/L std
5.00 mg/L std
1.00 mg/L std
0.500 mg/L std
0.100 mg/L std
0.000 mg/L std
ICV/LCSW1
ICB/PBW1
Sample A
Sample A Spike
Sample A Spike Dup (or Dup)
Sample B
etc.
CCV1 (after 10 sample injections)
CCB1

A CCV and CCB must follow the last sample.

5.2 Instrumental Conditions

5.2.1 Proportioning pump set to 35.

5.2.2 Reaction module 10-107-06-2-D

5.2.3 Injection valve with 50 cm sample loop.

5.2.4 Colorimeter with 660 nm filter and 10 mm flow cell.

5.2.5 Pump tubes required:

5.2.5.1 Diluent/Carrier: orange/orange

5.2.5.2 Buffer: blue/blue

5.2.5.3 NaOH: white/white

5.2.5.4 Sodium Salicylate: black/black

5.2.5.5 Hypochlorite: orange/white

5.3 Analytical Operation

5.3.1 Sample Digestion:

5.3.1.1 At block digestor: Set Temp1 to 160, Time1 to 070, Temp2 to 380, and Time2 to 120. Turn the block on.

5.3.1.2 Rinse sample digestion tubes 3x with DIW.

5.3.1.3 Place 20 mL of sample in tubes. (Less sample can be used if samples are suspected to be high.) For the ICB/PBW add 20 mL DIW. For ICV use less that 20 mL if necessary.

5.3.1.4 If necessary, dilute samples to 20 mL with DIW.

5.3.1.5 Add 2 boiling chips to each tube.

5.3.1.6 Add 7.5 mL digestion solution.

5.3.1.7 To MS and MSD add 2.0 mL 100. mg/L stock NH_3-N solution.

5.3.1.8 All one drop of antifoam to all the sample tubes. (More may be added during digestion if necessary.)

5.3.1.9 Wipe off the bottoms of the tubes with a towel.

5.3.1.10 When heating block is up to temp1 (first red light will be on) place sample racks in the block and press start once.

5.3.1.11 Rinse off cold fingers with DIW and place near digestor.

5.3.1.12 When timer goes off after 70 min, press start again and place cold fingers in sample tubes. Wait for 2 hours.

5.3.1.13 When timer goes off again, take tubes out off the digestor, take off cold fingers and let samples cool near a hood for five minutes. Rinse off cold fingers with DIW (be careful they will have residual acid on them).

5.3.1.14 To each sample tube add enough DIW to bring the volume to 20 mL. This usually takes 18.5 mL. Vortex the samples immediately.

5.3.1.15 Cover sample tubes and set aside until analysis.

5.3.2 Analysis:

5.3.2.1 Turn on Lachat and autosampler and allow to warm up for 10 minutes.

5.3.2.2 Download method by selecting "TKN-BD" in the Analysis Select and Download screen.

5.3.2.3 Lock pump tube cassettes down on the pump and raise the tension levers to vertical.

5.3.2.4 Place pump tubes in DIW and check for leaks or air bubbles.

5.3.2.5 Place pump tubes in the appropriate reagent containers except for the salicylate. Leave that tube in the DIW until all the other reagents have flowed through the system, then place it in the reagent bottle.

5.3.2.6 Make working standards as described in 2.2.3.

5.3.2.7 Begin calibration by choosing "Calibrate Now" under the "Submit" menu. Follow instructions on the screen to begin calibration.

5.3.2.8 Load samples into tray and record their positions in the "Identification" field under the "Edit" menu. Record any external dilutions under the "Dilution" field and the initial sample volumes under the "User Data 1" field.

5.3.2.9 Start the analysis by choosing "Submit Current Tray" field under the "Submit" menu. Fill in tray information when appropriate.

5.3.2.10 After analysis is complete, go to the "Results/Approval" screen. Save both the calibration in an excel format by choosing: Tray select/Calibration Tray/Tray name /Report/Choose Report Definition/excelltkn/Save Report. Save the sample data following the same procedure but choose "Sample Tray" instead of "Calibration Tray".

5.3.2.11 To shut down the instrument, take the pump tubes out of all reagent bottles and put them in the wash container (DIW), except the buffer. When the other reagents have been rinsed out of the system take the buffer pump tube out of the reagent bottle and put it in the wash container. Pump for five minutes to clean out the system.

5.3.2.12 Remove the pump tubes from the wash and let air pump through the system until the dry. Unlatch pump clips and turn off Lachat and autosampler.

5.4 Compound Quantification

5.4.1

$$\text{TKN-N, mg/L} = \frac{\text{mg/L in digest} * \text{dilution}}{\text{mls of sample}}$$

6. Reports

6.1 Data Packet Organization

6.1.1 A data package will consist of a copy of the Excel spreadsheet and a copy of the QC-DB report form.

6.2 Quality Control Reports

6.2.1 All results for quality control tests are entered into the lab data base using the QC_DB program. The routine minimum is a method blank report, an MS/MSD or MS/duplicate report, and an SRM, blank spike or other report.

6.3 Sample Result Reports

6.3.1 Data Qualifying Flags

6.3.1.1 Sample report results are qualified with data qualifying flags. These flags have the following definitions:

CODE	Definition
U :	The analyte of interest was not detected, to the limit of detection indicated.

Appendix I

QC Summary Table
Laucks Testing Laboratories
Method __351.2_____ QA Requirements and Corrective Actions

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Initial Calibration	3 pt. calibration	5 pt. calibration R≥0.995	Daily	Recalibrate	Copy of calibration in package
Initial Calibration Verification/ SRM	90-110%	90-110% or manufactures limits	At the beginning of the run	Recalibrate	Included in raw data
Initial Calibration Blank	Following calibration	Less than the reporting limit	At the beginning of the run	Reanalyze or recalibrate	Included in raw data
Continuing Calibration Verification	90-110%	90-110%	After every 10 samples and the last sample	Reanalyze or recalabrate	Included in raw data
Continuing Calibration Blank	After every 10 samples and the last sample	Less than the reporting limit	After every 10 samples and the last sample	Reanalyze or recalibrate	Included in raw data
Method Blank	1 per batch <MDL	Less than the reporting limit	1/20	Discuss with the QC Officer. May require reanalysis, or redigestion.	Included in raw data
Matrix Spike Recovery	90-110% 10% of samples	See QC database	1/20	Reanalyze	Included in raw data
MS/MSD RPD	None required	See QC database	1/20	Reanalyze	Included in raw data
Duplicate % Difference	None required	See QC database	1/20	Reanalyze	Included in raw data

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1.0 PURPOSE

The purpose of this document is to specify a consistent procedure for the quality assurance review of electronic and hard copy data bases. This SOP outlines the requirements for establishment of a Data Base Record File, Quality Assurance review procedures, and documentation of the Quality Assurance Review Process.

2.0 SCOPE

The methods described in this Standard Operating Procedure (SOP) shall be used consistently for all projects managed by personnel located in the Northeast Region of Brown & Root Environmental (Pittsburgh, PA; Wayne, PA; Wilmington, MA; and Holt, MI), for any large contracts managed by the Northeast Region (e.g., NORTHDIV CLEAN, SOUTHDIV CLEAN, ARCS I, ARCS III, etc.), and by other offices of Brown & Root Environmental at the discretion of the Project Manager. Smaller projects (as determined by Project Manager) are outside the scope of this SOP.

3.0 GLOSSARY

Chain-of-Custody Form - A Chain-of-Custody Form is a printed form that accompanies a sample or a group of samples from the time of sample collection to the laboratory. The Chain-of-Custody Form is retained with the samples during transfer of samples from one custodian to another. The Chain-of-Custody Form is a controlled document that becomes part of the permanent project file. Chain-of-Custody and field documentation requirements are addressed in SOP SA-6.1.

Electronic Data Base - A database provided on a 5.25" or 3.5" diskette or a laser disk. Such electronic data bases will generally be prepared using public domain software such as DBase, RBase, Oracle, Visual FoxPro, Microsoft Access, Paradox, etc.

Hardcopy Database - A printed copy of a data base prepared using the software discussed under the definition of an electronic data base.

Sample Tracking Summary - A printed record of sample information including the date the samples were collected, the number of samples collected, the sample matrix, the laboratory to which the samples were shipped, the associated analytical requirements for the samples, the date the analytical data were received from the laboratory, and the date that validation of the sample data was completed.

4.0 RESPONSIBILITIES

Database Records Custodian - It shall be the responsibility of the Database Records Custodian to update and file the Sample Tracking Summaries for all active projects on a weekly basis. It shall be the responsibility of the Database Records Custodian to ensure that the most recent copies of the Sample Tracking Summaries are placed in the Database Records file. It shall be the responsibility of the Database Records Custodian to ensure that a copy of all validation deliverables is provided to the Project Manager (for placement in the project file). It shall be the responsibility of the Database Records Custodian to ensure that photocopies of all validation deliverables and historical data and reports (as applicable) are placed in the Database Records file.

Data Validation Coordinator - It shall be the responsibility of the Data Validation Coordinator (or designee) to ensure that the Sample Tracking Summaries are maintained by the Database Records Custodian. It shall be the responsibility of the Data Validation Coordinator (or designee) to ensure that

photocopies of all data validation deliverables are placed in the applicable Database Records file by the Database Records Custodian.

Earth Sciences Department Manager - It shall be the responsibility of the Earth Sciences Department Manager (or equivalent) to ensure that all field personnel are familiar with the requirements of this Standard Operating Procedure (specifically Section 5.5).

Field Operations Leader - It shall be the responsibility of the Field Operations Leader of each project to ensure that all field technicians or sampling personnel are thoroughly familiar with this SOP, specifically regarding provision of the Chain-of-Custody Forms to the Database Records Custodian. Other responsibilities of the Field Operations Leader are described in Sections 5.4 and 5.5.

Information Management Systems Manager - It shall be the responsibility of the Information Management Systems Manager to ensure that copies of original electronic deliverables (diskettes) are placed in both the project files and the Database Records File. It shall be the responsibility of the Information Management Systems Manager (or designee) to verify the completeness of the database (presence of all samples) in both electronic and hardcopy form in the Database Records File. It shall be the responsibility of the Information Management Systems Manager (IMSM) to ensure that Quality Assurance Reviews are completed and are attested to by Quality Assurance Reviewers. It shall be the responsibility of the Information Management Systems Manager to ensure that records of the Quality Assurance review process are placed in the Database Records File. It shall be the responsibility of the Information Management Systems Manager to ensure that both electronic and hardcopy forms of the final data base are placed in both the project and the Database Record File. It shall be the responsibility of the Information Management Systems Manager to ensure that data validation qualifiers are entered in the data base.

Furthermore, it shall be the responsibility of the IMSM to participate in project planning at the request of the Project Manager, specifically with respect to the generation of level of effort and schedule estimates. To support the project planning effort, the IMSM shall provide a copy of the database checklist included as Attachment A to the project manager. It shall be the responsibility of the IMSM to generate level of effort and budget estimates at the time database support is requested if a budget does not exist at the time of the request. The database checklist shall be provided to the Project Manager at the time of any such requests. It shall be the responsibility of the IMSM to notify the Project Manager of any anticipated level of effort overruns or schedule noncompliances as soon as such problems arise along with full justification for any deviations from the budget estimates (provided they were generated by the IMSM). It shall be the responsibility of the IMSM to document an changes to the scope of work dictated by the Project Manager, along with an estimate of the impact of the change on the level of effort and the schedule.

Program/Department Managers - It shall be the responsibility of the Department and/or Program Managers (or designees) to inform their respective department's Project Managers of the existence and requirements of this SOP.

Project Manager - It shall be the responsibility of each Project Manager to determine the applicability of this SOP based on: (1) program-specific requirements, and (2) project size and objectives. It shall be the responsibility of the Project Manager (or designee) to ensure that the Field Operations Leader is familiar with the requirements regarding Chain-of-Custody Form provision to the Data Base Records Custodian. It shall be the responsibility of the Project Manager (or designee) to determine which, if any, historical data are relevant and to ensure that such data (including all relevant information such as originating entity, sample locations, sampling dates, etc.) are provided to the Database Records Custodian for inclusion in the Database Records File. It shall be the responsibility of the Project Manager

to obtain project planning input regarding the level of effort and schedule from the Information Management System Manager. It shall be the responsibility of the Project Manager to complete the database checklist (Attachment A) to support the level of effort and schedule estimate and to facilitate database preparation and subroutine execution.

Risk Assessment Department Manager - It shall be the responsibility of the Risk Assessment Department Manager to monitor compliance with this Standard Operating Procedure, to modify this SOP as necessary, and to take corrective action if necessary. Monitoring of the process shall be completed on a quarterly basis.

Quality Assurance Reviewers - It shall be the responsibility of the Quality Assurance Reviewers to verify the completeness of the sample results via review of the Chain-of-Custody Forms and Sample Tracking Summaries. It shall be the responsibility of the Quality Assurance Reviewers to ensure the correctness of the data base via direct comparison of the hardcopy printout of the data base and the hardcopy summaries of the original analytical data (e.g., Form Is provided in data validation deliverables). Correctness includes the presence of all relevant sample information (all sample information fields), agreement of the laboratory and database analytical results, and the presence of data validation qualifiers.

Quality Manager - It shall be the responsibility of the Quality Manager to monitor compliance with this Standard Operating Procedure via routine audits.

5.0 PROCEDURES

5.1 Introduction

Verification of the accuracy and completeness of an electronic data base can only be accomplished via comparison of a hardcopy of the database with hardcopy of all relevant sample information. The primary purposes of this SOP are to ensure that 1) all necessary hardcopy information is readily available to Quality Assurance Reviewers; 2) ensure that the Quality Assurance review is completed in a consistent and comprehensive manner, and; 3) ensure that documentation of the Quality Assurance review process is maintained in the project file.

5.2 File Establishment

A Database Record file shall be established for a specific project at the discretion of the Project Manager. Initiation of the filing procedure will commence upon receipt of the first set of Chain-of-Custody documents from a Field Operations Leader or sampling technician. The Database Record Custodian shall establish a project-specific file for placement in the Database Record File and will ensure that no information is removed from the file without the use of an "outcard." Each file in the Database Record File shall consist of standard components placed in the file as the project progresses. Each file shall be clearly labeled with the project number, which shall be placed on the front of the file drawer and on each and every hanging file folder relevant to the project. The following constitute the minimum components of a completed file:

- File Index
- Electronic Deliverables
- Sample Tracking Forms
- Chain-of-Custody Forms
- Data Validation Letters
- Historical Data (If applicable)

- Final Electronic Data Base
- Final Hardcopy Data Base
- Quality Assurance Records

Each file in the Database Record File must have an index summarizing the contents of the file. It shall be the responsibility of the Database Record Custodian to maintain the file index such that it is always current. The file index should specifically list the content of each of the subsections of the file and must also summarize the Sample Delivery Group numbers and samples and associated analyses associated with each Sample Delivery Group. Additional file requirements as well as database quality assurance procedures are summarized in the remainder of this section.

It should be noted that the majority of this "duplicate" file (i.e., the data validation deliverables) are already maintained by the Chemistry/Toxicology/Risk Assessment Department. Therefore, inclusion of the additional information outlined in this SOP is inconsequential from a cost perspective.

5.3 Electronic Deliverables

The format of electronic deliverables shall be specified in the laboratory procurement specification and shall be provided by the laboratory. The integrity of all original electronic data deliverables shall be maintained. This shall be accomplished via the generation of copies of each electronic deliverable provided by the laboratory. The original electronic deliverable shall be provided to the project manager for inclusion in the project file. A copy of the original electronic deliverable shall be placed in the Database Record File. The second copy shall be maintained by the Information Management Systems Manager (or designee) to be used as a working copy. The original and Database Record File copy of the electronic deliverable shall be converted to read only files by the Information Management Systems Manager or designee.

5.4 Sample Tracking Forms

Updated versions of the sample tracking form for each relevant project shall be maintained by the Database Record Custodian. The Sample Tracking Forms shall be updated any time additional Chain-of-Custody Forms are received from a Field Operations Leader or sampling technician, or at any time that data are received from a laboratory, or at any time that validation of a given data package (sample delivery group) is completed. The Data Validation Coordinator shall inform the Database Record Custodian of the receipt of any data packages from the laboratory and of completion of validation of a given data package to facilitate updating of the Sample Tracking Form. The Database Record Custodian shall place a revised copy of the Sample Tracking Form in the Database Record File anytime it has been updated. Copies of the updated Sample Tracking Form shall also be provided to the project manager to apprise the project manager of sample package receipt, completion of validation, etc.

5.5 Chain-of-Custody Forms

The Chain-of-Custody Forms for all sampling efforts will be used as the basis for (1) updating the Sample Tracking Form, and (2) confirming that all required samples and associated analyses have been completed. It shall be the responsibility of the Field Operations Leader (or sample technician) to provide a photocopy of all Chain-of-Custody Forms to the Database Record Custodian immediately upon completion of a sampling effort. The Database Record Custodian shall then place the copies of the Chain-of-Custody Form(s) in the Database Record File. Upon receipt of a sample data package from an analytical laboratory, the Data Validation Coordinator shall provide a copy of the laboratory Chain-of-Custody Form to the Database Record Custodian. The Database Record Custodian shall use this copy to update the Sample Tracking Summary and shall place the copy of the laboratory-provided Chain-of-

Custody Form in the Database Record File. The photocopy of the laboratory-provided Chain-of Custody Form shall be stapled to the previously filed field copy. Upon receipt of all analytical data, two copies of the Chain-of-Custody will therefore be in the file. Review of the Chain-of-Custody Forms will therefore be a simple mechanism to determine if all data have been received. Chain-of-Custody is addressed in SOP SA-6.1.

5.6 Data Validation Letters

All data validation deliverables (or raw data summaries if validation is not conducted) shall be provided for inclusion in both the Database Record File and the project file. If USEPA regional- or client-specific requirements are such that Form Is (or similar analytical results) need not be provided with the validation deliverable, copies of such results must be appended to the deliverable. It is preferable, although not essential that the validation qualifiers be hand-written directly on the data summary forms. The data validation deliverables (and attendant analytical summaries) will provide the basis for direct comparison of the database printout and the raw data and qualifiers.

5.7 Historical Data

At the direction of the Project Manager, historical data may also be included in a project-specific analytical data base. In the event that historical data are germane to the project, hardcopy of the historical data must be included in the Database Record File. Historical data may be maintained in the form of final reports or as raw data. The information contained in the historical data file must be sufficient to identify its origin, its collection date, the sample location, the matrix, and any and all other pertinent information. All available analytical data, Chain-of-Custody Forms, boring logs, well construction logs, sample location maps, shall be photocopied by the Project Manager (or designee) and placed in one or more 3-ring binders. All information shall be organized chronologically by matrix. It shall be the responsibility of the Project Manager (or designee) to ensure that all inconsistencies between analytical data, Chain-of-Custody Forms, boring logs, sample log sheets, and field logbooks are identified and corrected. The Project Manager (or designee) shall decide which nomenclature is appropriate and edit, initial and date all relevant forms. Data entry may only be performed on information that has undergone the aforementioned editing process, thereby having a direct correlation between hardcopy information and what will become the electronic database.

Sample spreadsheets shall be generated for all samples previously collected at the site (see Attachment B). The sample spreadsheets shall have specific references to all source documents. If many historical reports exist, the Project Manager shall maintain an organized library with outcards for tracking purposes.

5.8 Final Electronic Data Base

The final electronic database shall be filed in both the project and Database Record Files on diskettes, tapes, laser disks, etc. The final files shall be toggled as read only files. It shall be the responsibility of the Information Management Systems Manager to ensure that the final electronic files are provided to both the project and Database Record Files. The final electronic database shall be clearly labeled with the Site Name, Project Number, and Project Title, and shall indicate that the diskette, tape, or CD ROM is the final database including the completion date. An example is as follows:

MCAS Cherry Point
Project Number
RCRA Facility Investigation
Operable Unit 3
Final Electronic Database
Completed 06/15/96

5.9 Final Hardcopy Data Base

The final hardcopy data base shall be filed in both the project and Database Record Files as legible, reproducible printouts. The final database printouts shall be clearly identified as such on the cover page(s). It shall be the responsibility of the Information Management Systems Manager to ensure that the final hardcopy of the database are provided to both the project and Database Record Files.

The final hardcopy database shall be clearly labeled with the Site Name, Project Number, and Project Title, and shall indicate that the hardcopy is the final database including the completion date. An example is as follows:

MCAS Cherry Point
Project Number
RCRA Facility Investigation
Operable Unit 3
Final Hardcopy Database
Completed 06/15/96

The final hardcopy database must also clearly display an attestation that Quality Assurance review has been completed. Specifically, the signature of the Information Management Systems Manager (or designee must appear on the final hardcopy. The date of the final review and an attestation that the final review was completed must be provided. The attestation shall take the following form:

"Final Database Quality Assurance Review Completed By: _____ on __/__/__."

In addition, the final hardcopy database should reference the electronic database via reference to the medium used for storage (e.g, diskettes 1 through 3), and the final file names for all components of the database.

5.10 Quality Assurance Procedures

The Information Management System Manager (or designee) shall assign one or more individuals (Quality Assurance Reviewers) to complete Quality Assurance Review of the data base, either in its entirety or on an Sample Delivery Group-specific basis. Such review shall focus on the "accuracy" of the analytical results (do the numerical values agree with the results as provided by the laboratory), have the data validation qualifiers (if applicable) been entered and are they correct (i.e., do they agree with the qualifiers contained in the data validation letters), are all requested analytical results present in the Sample Delivery Group(s) or the database, are all required data base fields provided (e.g., northing, easting, sample depth, sampling date, matrix, site name, operable unit, total versus dissolved metals, sampling round, descriptive information such as "upstream", "downstream", "composite", and any other relevant information deemed appropriate by the Project Manager), are units provided and are they the correct units, are any fractions that were not analyzed in specific samples identified as such, does the data base indicate that validation has been completed, are all validation qualifiers defined in footnotes, are the results for duplicate samples and the average values for duplicates provided if required on a project-

specific basis, is the hardcopy database legible and suitable for photocopying if necessary, are quantitation or method detection limits provided for nondetected analytes, are the sample results segregated by matrix, are the Quality Control sample results provided. Additional requirements may be specified by the Project Manager during database task commencement. Upon completion of such Quality Assurance review, the Quality Assurance Reviewer shall attest that the review has been completed via the following statements:

"Intermediate Database QA Review Completed By: _____ on __/__/__."

"Data correct as provided in the attached summary." or

"Data incorrect as provided in the attached summary. Submitted for correction."

Copies of such intermediate database reviews shall be placed in the Database Record File. Upon completion of the intermediate database review, the database shall be returned for correction (if necessary) by the ISMS (or designee), and shall undergo an additional Quality Assurance review to ensure that all necessary modifications have been made. Upon satisfactory completion of the revision process, the final attestation discussed in Section 5.9 must be printed on the final data base.

5.11 Quality Assurance Records

Quality Assurance records for the Database Record File include the intermediate and final attestation copies discussed in the preceding two sections.

6.0 RECORDS

Records regarding database preparation and quality assurance review include all those identified in the previous section. Upon completion of the database task, records from the file will be forwarded to the Project Manager for inclusion in the project file, or will be placed in bankers boxes (or equivalent) for storage. The final records for storage shall include the following minimum information on placards placed on both the top and end of the storage box:

Database Record File
PROJECT NUMBER: ____
SITE NAME: _____
DATE FILED: __/__/__
SUMMARY OF CONTENTS ENCLOSED
BOX _ OF _

Project- or program-specific record keeping requirements shall take precedence over the record keeping requirements of this SOP.

ATTACHMENT A

DATABASE PLANNING CHECKLIST

PROJECT NAME _____ **PROJECT NUMBER** _____

PROJECT MANAGER _____ **PLANNING DATE** _____

1. Provide a general description of the project (regulatory authority, media to be samples, approximate number of samples by media, analyses by media, data evaluation tasks required):

_____

2. Provide a general description of the sample nomenclature that will be used for samples collected by Brown & Root Environmental:

_____

3. Will historical data be entered in the database? Yes No

4. Will historical data be used to define the nature and extent of contamination? Yes No

5. Will historical data be used for risk assessment purposes? Yes No

6. How much historical data exists (i.e., number of samples by matrix, analysis by matrix)?

_____

7. In what format will the historical data be provided? Hardcopy Electronic

8. If historical data are in electronic form, what software was used and what is the format?

_____

9. If historical data are in hardcopy form, will Form I's, summary tables, or reports be provided? Copies of historical data will be necessary to generate a budget estimate.

_____

10. Will Quality Assurance review of historical data be necessary? Yes No

ATTACHMENT A (Continued)
DATABASE PLANNING CHECKLIST

11. If Quality Assurance review of historical data is necessary, describe the scope of the Quality Assurance review:

_____
_____
_____
_____

12. Will a GIS database be necessary for the project? Yes No

    If so, Design Engineering should be consulted for a budget estimate.

13. What nomenclature has been (will be) used to identify field duplicate samples?

_____
_____
_____
_____

14. Will field duplicate results be averaged and presented as one result in the data base? Will they be presented as distinct results, or will both the average and the distinct results be presented?

_____
_____
_____
_____

15. How will the average value for duplicate samples be determined on a matrix-specific basis?

_____
_____
_____
_____

16. Are any unvalidated data to be included in the database? Yes No
17. Will unvalidated data be used for defining the nature and extent of contamination? Yes No
18. Will unvalidated data be used for risk assessment purposes? Yes No
19. Are any field screening data to be included in the database? Yes No
20. Will field screening data be used for defining the nature and extent of contamination? Yes No
21. Will field screening data be used for risk assessment purposes? Yes No
22. Will statistical correlation of laboratory and field screening data be necessary? Yes No
23. If a correlation exists between field screening and laboratory data, will the results of regression analysis be used to define nature and extent? Yes No
24. If a correlation exists between field screening and laboratory data, will the results of regression analysis be used to support the risk assessment? Yes No
25. Will field parameters be included in the database (e.g., pH, conductance, temperature)? Yes No
26. Will statistical correlations be necessary for TCLP versus RAS/SAS data? Yes No

ATTACHMENT A (Continued)
DATABASE PLANNING CHECKLIST

27. Will statistical correlations be necessary for filtered versus unfiltered samples? Yes No

28. Will any other statistical correlations be necessary? Yes No

29. Are there wells that have been screened in different aquifers? Yes No

30. Will data for various aquifers be segregated by depth? Yes No

31. Can the sample nomenclature system be used to identify wells in different aquifers? Yes No

32. Will samples from other matrices (soil, sediment, or surface water) be segregated by depth? Yes No

33. Can the sample nomenclature system be used to identify depth-specificity? Yes No

34. Have any removal actions be performed at the site? Yes No

    If removal actions have been performed, plan and cross-sectional views reflecting the extent of the removal action must be provided.

35. Will any composite sample results be included in the database? Yes No

36. If composite samples are included how will they be used for the nature and extent of contamination?

    _____

    _____

    _____

37. If composite samples are included how will they be used for the risk assessment?

    _____

    _____

    _____

38. Will the site be segregated into Areas of Concern, Solid Waste Management Units, etc? Yes No

39. Is the sample nomenclature adequate for such segregation? Yes No

    If the sample nomenclature is inadequate for assigning samples to an AOC or SWMU, the Project Manager or designee must provide a base map of tabular summary clearly delineating the relationship between each sample and each AOC/SWMU.

40. Were any temporal samples collected (e.g., quarterly sampling of wells)? Yes No

41. If temporal samples were collected, how will they be used to define the nature and extent of contamination?

    _____

    _____

    _____

42. If temporal samples were collected, how will they be used to support the risk assessment?

    _____

    _____

    _____

43. Are State, Federal, or Regional criteria to be included in data summary tables? Yes No

ATTACHMENT A (Continued)
DATABASE PLANNING CHECKLIST

44. Identify the criteria that must be presented in the summary tables.

_____
_____
_____

45. Will State, Federal, or Regional criteria be used to select COPCs? Yes No

46. Identify the criteria to be used as COPC selection tools.

_____
_____
_____

47. Are filtered and unfiltered surface water samples differentiated? Yes No

48. If such samples are differentiated, how?

_____
_____
_____

49. Which of these samples will be used for the human health risk assessment?

Surface Water	Filtered	Unfiltered
Groundwater	Filtered	Unfiltered

50. Which of these samples will be used for the ecological assessment?

Surface Water	Filtered	Unfiltered
Groundwater	Filtered	Unfiltered

51. Will background data be included in the database? Yes No

52. How are background samples identified?

_____
_____
_____

53. Will background results be used to support selection of COPCs? Yes No

54. What statistical analyses will be required for the background data?

_____
_____
_____

55. Will background data be segregated by depth? Yes No

56. What background matrices must be segregated by depth?

_____
_____
_____

ATTACHMENT A (Continued)
DATABASE PLANNING CHECKLIST

57. What format will be used for data presentation (e.g., appendices and summary tables, comprehensive text tables, tag maps, isoconcentration contours, etc.)?

_____

_____

_____

ATTACHMENT B
HISTORICAL DATA FOR _____
GROUNDWATER

Investigations	Investigative Well Identification	Installation Company & Date	Laboratory Parameters				
			09/82	11/82	01/83	02/83	04/83
Groundwater Monitoring Program (USACEWES, 1981).	WES-05-01-81	WES 09/14/81	C, D, E	B	F	A	
	WES-05-02-81	WES 09/15/81	C, D, E	B	F	A	
Hydrogeological Investigation of Waste Disposal Sites at the NSWC Crane, Indiana (Dunbar, 1982).	WE-05-03-81	WES 09/16/81	C, D, E	B	F	A	
	WES-05-04-81(*)	WES 10/01/81	C, D, E	B	F	A	
	05-04A (**)	WES 1986					
Definition of Contaminated Groundwater Plumes at Selected Waste Disposal Sites; Draft (Dunbar, 1984).	WES-05-05-81	WES 10/02/81	C, D, E	B	F	A	
	WES-05-06-81	WES 10/10/81	C, D, E	B	F	A	
	WES-05-07-81	WES 10/20/81	C, D, E		F	A	
	WES-05-08-81	WES 11/04/81	C, D, E	B	F	A	
U.S. Dept. of the Navy IRP RFI Phase III Groundwater Investigations for NSWC Crane, Indiana Old Burn Pit; U.S. Army Corps of Engineers; WES (June 1991).	WES-05-09-82	WES 10/27/82		B			
	WES-05-10-82	WES 10/27/82		B			
	WES-05-11-82	WES 10/29/82		B			
	WES-05-12-82	WES 10/30/82		B			
	WES-05-13-82	WES 11/01/82		B			
	WES-05-14-83	WES 01/10/83			B		
	WES-05-15-83	WES 01/11/83			B		G
	WES-05-16-83	WES 01/11/83			B		
	WES-05-17-83	WES 02/02/82			B		G
	WES-05-18-83	WES 02/03/83			B		G
	WES-05-19-83	WES 02/03/83			B		G

(*) Original contaminated well yielding highest concentrations of contaminants.
(**) Replacement well.
A Metals, chloride, specific conductance, TOC, phenols, sulfate, TOX, pH.
B VOCs.
C Metals, fluoride, nitrate-nitrate, pest., chlorinated herbicides, GRA/GRB, chloride, phenols, sulfate, pH specific conductance, TOC, TOX.
D GRA.
E GRB.
F Metals.

APPENDIX B

BROWN & ROOT ENVIRONMENTAL
AUDIT CHECKLIST

Tetra Tech NUS, Inc.

SOUTHDIV CLEAN Field Quality Assurance Audit

CTO 19 – Crane Landfarm

Site: _____

Location: _____

Audit Date(s): _____

Auditor: _____

Task Order Manager: _____

Field Operation Leader: _____

Site Safety Office: _____

Other TtNUS persons present on site:

_____

_____

_____

_____

_____

Subcontract personnel present on site:

_____

_____

_____

_____

Project Description: Redevelop and sample the 7 wells that were installed at this SWMU for volatile organics, mercury (total and dissolved), Appendix 9 metals (total and dissolved), cyanide, nitrate/nitrite, hexavalent chromium, ammonia, phosphorous, and total kjeldahl nitrogen. Sampling is to be conducted for 5 rounds. Information is required to complete the Phase II Groundwater RFI for this unit.

_____

_____

_____

_____

_____

FIELD DATA FILE REVIEW:

Project Planning Documents: Work Plan and QAPP

Current Field Forms:

Subcontract Specifications:

SAMPLING & ANALYSIS REVIEW:

Secure Sample Storage:

Task Modification Field Records Utilized:

Sample Custody:

Instrument Calibration: Check Hach Test Kits for hexavalent chromium analysis

Decontamination Fluids:

General Cleanliness:

Field Preservation:

Field/Site Log Book Review:

MEDIA-SPECIFIC SAMPLING PROCEDURES/EQUIPMENT:

Groundwater: Refer to Section 2.3 of the Work Plan for specific items to check

Notes: _____

IDW: _____

Air Monitoring: _____

QA Sampling/Frequency: _____

Equipment Storage: _____

Packaging & Shipping: _____

OTHER FIELD INVESTIGATION ACTIVITIES:

(Surveying, GPS, lab analysis, field screening, aquifer testing, etc.): _____

FIELD EQUIPMENT CONDITION:

(safety switches, ropes, cables, surveying, field analysis, screening, etc.):

IDW MANAGEMENT:

(Management, cleanliness, security, location, etc.): _____

DECONTAMINATION:

Solutions (solvents, acids, soap, water, etc.):

Sampling Equipment:

Decon Area:

Other:

ADDITIONAL COMMENTS:

RECOMENDATIONS:

RESOLUTION:

Auditor: _____ **Date:** _____

Field Operation Leader: _____ **Date:** _____

Task Order Manager: _____ **Date:** _____

Distribution:

Task Order Manager: _____

Program Deputy/PMO File: _____

QA Officer: _____

Other: _____