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Final
Quality Assurance Project Plan
for
Operable Unit 3
Remedial Investigation/Feasibility Study

Naval Industrial Reserve
Ordnance Plant
Fridley, Minnesota

Volume III of IV

Southern Division
Naval Facilities Engineering Command
Contract Number N62467-94-D-0888
Contract Task Order 0003

June 1997

FINAL
QUALITY ASSURANCE PROJECT PLAN
FOR
OPERABLE UNIT 3 REMEDIAL INVESTIGATION/FEASIBILITY STUDY

NAVAL INDUSTRIAL RESERVE ORDNANCE PLANT
FRIDLEY, MINNESOTA

VOLUME III OF IV

REVISION 2
JUNE 1997

COMPREHENSIVE LONG-TERM
ENVIRONMENTAL ACTION NAVY (CLEAN) CONTRACT

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PREFACE

This Quality Assurance Project Plan (QAPP) is Volume III of the four-volume Work Plan.

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ACRONYMS/ABBREVIATIONS

AA	Atomic Absorption
B&R Environmental	Brown & Root Environmental
BFB	Bromofluorobenzene
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CFU	Colony Forming Units
CLEAN	Comprehensive Long-Term Environmental Action, Navy
CLP	Contract Laboratory Program
COC	Chain of Custody
CRDL	Contract Required Detection Limit
CRQL	Contract Required Quantitation Limit
CVAA	Cold Vapor Atomic Absorption
DFTPP	Decaflourotriphenyl phosphine
DPT	Direct-Push Technology
DRO	Diesel Range Organics
DQO	Data Quality Objective
EC	Electron Capture
Fe(II)	Ferrous (Reduced) Iron
FFA	Federal Facilities Agreement
FOL	Field Operations Leader
FS	Feasibility Study
FSP	Field Sampling Plan
FTMR	Field Task Modification Request
GC	Gas Chromatograph
GFAA	Graphite Furnace Atomic Absorption
GRO	Gasoline Range Organics
HBV	Health Based Value
HRL	Health Risk Limit
ICP	Inductively Coupled Plasma
LCS	Laboratory Control Sample
LIMS	Laboratory Information Management System
MCL	Maximum Contaminant Level
MDL	Method Detection Limit
MPCA	Minnesota Pollution Control Agency
MS	Mass Spectrophotometer
MS/MSD	Matrix Spike/Matrix Spike Duplicate
NEESA	Naval Energy and Environmental Support Activity
NFESC	Naval Facilities Engineering Service Center
NIROP	Naval Industrial Reserve Ordnance Plant
NIST	National Institute of Science and Technology
NTU	Nephelometric Turbidity Units
ORP	Oxidation Reduction Potential
OU1	Operable Unit 1
OU2	Operable Unit 2
OU3	Operable Unit 3
PARCC	Precision, Accuracy, Representativeness, Comparability, Completeness
PCB	Polychlorinated Biphenyl
PQL	Practical Quantitation Limit
QA	Quality Assurance

ACRONYMS/ABBREVIATIONS (Continued)

QAM	Quality Assurance Manager
QAPP	Quality Assurance Project Plan
QC	Quality Control
QN	Quality Notice
RA	Risk Assessment
RAS	Routine Analytical Services
RI	Remedial Investigation
RPD	Relative Percent Difference
SDG	Sampling Delivery Group
SOP	Standard Operating Procedure
SOW	Statement of Work
SO_4^{2-}	Sulfate
SRM	Standard Reference Materials
TAL	Target Analyte List
TCL	Target Compound List
TOC	Total Organic Carbon
TSS	Total Suspended Solids
UCL	Upper Ninety-Five Percent Confidence Limit
U.S. EPA	United States Environmental Protection Agency
VOC	Volatile Organic Compound
%R	Percent Recovery

1.0 PROJECT DESCRIPTION

This project description outlines the overall scope of a Remedial Investigation (RI) to be performed for Operable Unit 3 of the Naval Industrial Reserve Ordnance Plant (NIROP) located in Fridley, Minnesota. Operable Unit 3 includes contaminant sources in all environmental media (soil and groundwater) at the site. The Quality Assurance Project Plan presents the organization, objectives, planned activities, and specific Quality Assurance/Quality Control (QA/QC) procedures associated with the Work Plan (and addenda) for the RI. 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. EPA Region V and Minnesota Pollution Control Agency requirements, regulations, guidance, and technical standards.

1.1 INTRODUCTION

This Quality Assurance Project Plan (QAPP) has been prepared by Brown & Root Environmental (B&R Environmental) on behalf of the United States Navy Southern Division Naval Facilities Engineering Command and the NIROP, Fridley, Minnesota. This QAPP and other associated documents, including Work, Field Sampling, and Health and Safety Plans constitute the project planning documents for the OU3 RI.

1.1.1 Overall Project Objectives

The general project objectives for the Fridley Operable Unit 3 (OU3) Remedial Investigation (RI) are outlined in the Federal Facilities Agreement (FFA) for the NIROP. Attachment A of the FFA outlines the general project objectives as follows:

> "(1) identify all source areas of contamination; (2) identify the extent and magnitude of soil, subsoil, surface water, and groundwater contamination; (3) gather all necessary data to support the Feasibility Study (FS) and Risk Assessment (RA), and; (4) provide information and data needed for the selection and implementation of response actions at the site."

The FFA goes on to state (Section IV, Task A, Part 2) that RI Work Plans (upon implementation) are intended to:

"(1) provide for the complete characterization of the site and its actual or potential hazard to public health, welfare and the environment; (2) produce sufficient data and information to allow the Navy to submit the review of Alternatives Report; and, (3) produce data of sufficient quantity and adequate technical content to assess possible alternative response actions during the FS."

These general project objectives, except for surface water evaluation which was addressed in the Operable Unit 1 (OU1) RI, have provided the basis for the development of specific Data Quality Objectives (DQOs), as discussed in Section 4.0 of the attendant Work Plan (Volume I), as well as the scope of work for the OU3 RI.

1.1.2 Project Status/Phase

The RI at NIROP Fridley has been undertaken on an operable unit basis. The first operable unit (OU1) included site groundwater and surface water. The OU1 RI was completed by RMT Inc. in June 1987. The second operable unit (OU2) included all facility soils (unsaturated zone) other than those beneath the plant building footprint. The RI for OU2 was completed by RMT Inc. in September, 1993. The third operable unit (OU3) includes potential source areas at the facility. This QAPP and all attendant project planning documents apply to OU3. OU2 has been made a subset of OU3. All conclusions from the OU2 RI will be included in the OU3 FS.

The OU3 RI will be completed in a phased manner. The first phase will include chemical and physical characterization of the soils and shallow groundwater beneath the plant. The second phase will include additional groundwater characterization to delineate potential contaminant migration associated with any potential source areas identified during Phase I.

1.1.3 QAPP Preparation Guidelines

This Quality Assurance Project Plan has been prepared in accordance with the general guidance outlined in the United States Environmental Protection Agency (U.S. EPA) Region V Model Superfund Quality Assurance Project Plan dated January 1996. Additional guidance regarding the QAPP contents was provided by representatives of U.S. EPA Region V and the Minnesota Pollution Control Agency (MPCA) during a teleconference held on February 20, 1996. Representatives of U.S. EPA Region V, the MPCA, the United States Navy Naval Facilities Engineering Command (Southern Division), and B&R Environmental participated in the teleconference.

1.2 FACILITY DESCRIPTION

A brief discussion of the NIROP, including its location, size and borders, regional geology, hydrogeology, hydrology, topography, etc. is provided in the remainder of this section. The majority of this information is contained in the Work Plan for the OU3 RI, and specific sections of the Work Plan are referenced as appropriate.

1.2.1 Location

The NIROP Fridley is located approximately 700 feet east of the Mississippi River in the City of Fridley, Anoka County, Minnesota. A site location map for the facility is provided as Figure 2-1 of the Work Plan for the Naval Industrial Reserve Ordnance Plant (Volume I).

1.2.2 Facility Size and Borders

The NIROP encompasses approximately 83 acres. The facility is bordered on the east by the Burlington Northern rail yard, on the north by various industrial facilities, on the west by East River Road, and on the south by United Defense, LP.

1.2.3 Topography

The NIROP is located on a broad, flat, alluvial terrace of the Mississippi River at an elevation of approximately 835 feet (National Geodetic Vertical Datum of 1929). Slopes across the site are five percent or less (Envirodyne Engineers, Inc., June 1983 Initial Assessment Study, Naval Industrial Reserve Ordance Plant, Minneapolis, Minnesota).

1.2.4 Local Hydrology and Hydrogeology

Detailed information regarding regional geology, hydrogeology, and hydrology are provided in Sections 2.7.1, 2.7.2, and 2.7.3 of the Work Plan (Volume I).

1.3 FACILITY HISTORY

Detailed discussions of the general history of the NIROP and past data collection activities at the facility are included in the Work Plan (Volume I). Specific sections of the Work Plan are cited and incorporated by reference in the remainder of this section, as applicable.

1.3.1 General History

The NIROP has been in operation since 1940 and is a production facility for Naval ordnance. Items produced at the facility have included gun mounts and advanced missile launching systems. Additional general background regarding historical industrial activities at the NIROP is provided in Section 2.4 of the Work Plan for the facility (Volume I).

1.3.2 Past Data Collection Activities

A chronological history of events at the NIROP, including past data collection activities is provided in Section 2.5 of the Work Plan (Volume I). A concise summary of historical events including previous investigations is provided in Table 2-1 of the Work Plan (Volume I).

1.3.3 Current Status

At the current time, the Remedial Investigations for Operable Units 1 and 2 have been completed. A feasibility study of alternatives was completed for Operable Unit 1, and, as a result of the feasibility study, a Record of Decision was signed requiring implementation of a containment system (active pumping) to prevent continued offsite migration of TCE in groundwater. Operable Unit 3 includes potential source areas at the facility.

1.4 PROJECT OBJECTIVES

This section outlines the overall project objectives for the OU3 RI at NIROP, Fridley. 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. Data Quality Objectives are developed in Section 1.4.3.

1.4.1 Specific Objectives and Associated Tasks

Four primary objectives, as discussed in Section 1.1.1, are identified for the NIROP Fridley OU3 RI. A phased investigation will satisfy these objectives as previously discussed in Section 1.1.2. The specific objectives for each phase of the OU3 RI are outlined in the following subsections.

1.4.1.1 Phase I

Characterize the soils beneath the production facility from the standpoint of potential direct contact impacts on human health (utility and construction workers) under existing site conditions and under a benchmark future condition (industrial land use).

Characterize the soils beneath the production facility from the standpoint of potential sources of groundwater contamination. Groundwater contamination is considered any concentration exceeding a U.S. EPA Maximum Contaminant Level (MCL) or a MPCA Health Risk Limit (HRL). The more conservative value (MCL or HRL) will be used. If neither a MCL nor a HRL exist for a parameter, then a state Health Based Value (HBV) will apply.

Characterize stratigraphy to define potential preferential flow conduits for groundwater contamination and/or dense non-aqueous phase liquids.

Tasks necessary to accomplish the objectives of Phase I include the collection of near-surface and subsurface soil samples, and shallow groundwater samples from beneath the building footprint.

1.4.1.2 Phase II

Characterize stratigraphy and groundwater beneath the production facility in order to locate contaminant sources and obtain information needed to evaluate remediation alternatives.

Tasks necessary to accomplish the objectives of Phase II include the installation of groundwater monitoring wells within the building, and collection and analysis of groundwater samples from the newly installed wells.

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 OU3 RI. 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 the completion of soil borings, installation and development of monitoring wells, and groundwater sampling and analysis. Field measurements will include only those completed using simple field instrumentation, field test kits, a portable colorimeter, and a field gas chromatograph (GC).

Field measurements of total volatile organics will be completed using a Photoionization Detector or Flame Ionization Detector. These measurements will be used to determine appropriate subsurface sample horizons to be submitted for laboratory analysis and in safety monitoring to determine breathing zone conditions for site workers.

Field parameters including pH, specific conductance, turbidity, dissolved oxygen, and temperature will be completed for all aqueous phase samples using a water quality meter as discussed in Section 7.5.2 of the FSP. These measurements will be used to support monitoring well development and purging of stagnant water from well casings. Specific conductance and pH will also be used as general indicators of water quality.

Additional water parameters will include oxidation-reduction potential (ORP), dissolved ferrous (reduced) iron, dissolved reduced manganese, and hydrogen sulfide (as sulfide). (Note that ORP is sometimes referenced as Eh.) These measurements, along with pH, dissolved oxygen, temperature, and several laboratory parameters, will be used to assess the natural attenuation of chlorinated solvents in the groundwater system at the NIROP Fridley. In addition to measurement using the water quality meter, dissolved oxygen will also be measured using a field test kit. ORP will be measured using a water quality meter. The remaining field parameters (dissolved ferrous iron, dissolved reduced manganese, and hydrogen sulfide) will be measured using a portable colorimeter. Tables 1-1 and 1-2 provide a summary of field parameters and associated ranges and increments of detection.

TABLE 1-1

RANGES AND INCREMENTS OF DETECTION FIELD PARAMETERS FOR GROUNDWATER QUALITY TESTING NIROP FRIDLEY, MINNESOTA

Parameter	Method	Range/Increment
pH	meter	0-14 / 0.01 units
Specific Conductance	meter	0-100 / 0.01 millimhos/cm
Turbidity	meter	0-800 / 0.1-1 NTU
Dissolved Oxygen	meter	0-19.9 / 0.01-0.1 mg/L
Temperature	meter	0-50 / 0.1-1°C

TABLE 1-2

ANALYTICAL DETECTION LIMITS
FIELD PARAMETERS FOR EVALUATION OF NATURAL ATTENUATION
NIROP FRIDLEY, MINNESOTA

Parameter	Method	Range / Increment
pH	meter	0 to 14 / 0.01 units
Dissolved Oxygen	modified Winkler titration test kit	0.02 to 10 / 0.02 mg/L
Temperature	meter	-5.0 to 50 / 0.4°C
Oxidation Reduction Potential	meter	-1500 to 1500 mV / 2% of reading plus 1 count
Dissolved Ferrous Iron	10-phenanthroline method(1)	0 to 5 / 0.01 mg/L
Dissolved Reduced Manganese	PAN method(1) Periodate oxidation method(1)	0 to 0.8 / 0.001 mg/L 0 to 20 / 0.1 mg/L
Hydrogen Sulfide (as sulfide)	Methylene Blue Method(1)	0 to 0.6 / 0.001 mg/L

(1) Portable colorimeter

On-site analysis of soil samples will also be performed using a field GC for the volatile organic compounds shown in Table 1-3. As further discussed in Section 7.3.1 of the FSP, soil samples will be collected at 4-foot intervals (using direct-push technology or DPT) or 5-foot intervals (during installation of permanent monitoring wells) down to the termination depths of the borings for purge-and-trap field GC analysis. The results of these analyses will be used to evaluate the vertical distribution of contaminated soil during drilling, to guide the sampling effort, and to quantitatively evaluate the protection of groundwater. The soil sample with the highest field GC result in the 2- to 12-foot interval, as well as the soil sample from the 0- to 2-foot interval, of the DPT borings will be collected and submitted for analysis by the fixed-base laboratory.

1.4.2.2 Laboratory Parameters

Laboratory parameters will include Target Compound List (TCL) volatile and semivolatile organics and polychlorinated biphenyls, Target Analyte List (TAL) metals and cyanide, hexavalent chromium, sulfate, total suspended solids (TSS), total hardness (as $CaCO_3$), alkalinity (as $CaCO_3$), nitrate, nitrite, dissolved chloride, dissolved bromide, dissolved phosphate, dissolved methane, and total organic carbon (TOC). Pyridine will also be added to the semivolatile Target Compound List since this compound was a constituent in some of the products used at the site. Total hardness, TSS, and alkalinity will be used for engineering analysis during the feasibility study. The TCL and TAL compounds/analytes will be used to support decision making via direct comparison with the preliminary health-based numeric decision rules outlined in Section 4.0 of the Work Plan. It should be noted that SW-846 Method 8260A, modified by using a 25 mL purge volume, will be used for the analysis of TCL volatiles for groundwater samples. This method will be used in place of standard Contract Laboratory Program (CLP) protocol in order to achieve lower quantitation limits for volatile organic compounds in groundwater, since these are the compounds of primary concern at the site. This low-concentration method will also be used for the analysis of all trip blanks associated with the OU3 investigation. Analytical methods are further discussed in Section 7. Representative soil samples will be analyzed for hexavalent chromium to evaluate the speciation of total chromium. As detailed in Standard Operating Procedure LTL-7014 (Appendix A) matrix spike results of the hexavalent chromium analyses may also necessitate fixed-base laboratory analysis of soil samples for pH, ORP, ferrous iron, and sulfides. Based on holding time requirements, as presented in Table 4-1 of the FSP, analyses for these four parameters will be performed immediately upon receipt by the laboratory for each hexavalent chromium sample designated for matrix spike analysis. Representative soil samples will be analyzed for TOC to evaluate the availability of carbon sources for bioremediation options. The remaining parameters (sulfate, nitrate, nitrite, dissolved chloride, dissolved bromide, dissolved phosphate,

TABLE 1-3

PRACTICAL QUANTITATION LIMITS - FIELD GC VOLATILE ORGANIC ANALYSIS NIROP FRIDLEY, MINNESOTA

Parameter	Soil Samples
	PQL(1)
Volatile Organic Compounds	µg/kg
Acetone	5
Benzene	1
Bromoform	1
Bromomethane	5
2-Butanone	5
Carbon disulfide	1
Carbon tetrachloride	1
Chlorobenzene	1
Chloroethane	5
Chloroform	1
Chloromethane	5
1,1-Dichloroethane	1
1,2-Dichloroethane	1
1,1-Dichloroethene	1
cis-1,2-Dichloroethene	1
trans-1,2-Dichloroethene	1
Ethylbenzene	1
2-Hexanone	5
4-Methyl-2-pentanone	5
Methylene chloride	1
Styrene	1
1,1,2,2-Tetrachloroethane	1
1,1,1-Trichloroethane	1
1,1,2-Trichloroethane	1
Trichloroethene	1
Tetrachloroethene	1
Toluene	1
Vinyl chloride	5
m,p-Xylenes	1
o-Xylene	1

1 PQL Practical Quantitation Limit.

and dissolved methane) will be used in conjunction with the field parameters previously discussed to assess the natural attenuation of chlorinated solvents in the groundwater system at the NIROP Fridley. Tables 1-4 through 1-6 provide a summary of all target laboratory analytes and associated Contract Required Quantitation and Method Detection Limits (TCL organics via CLP protocol), Contract Required and Instrument Detection Limits (TAL inorganics), and Practical Quantitation and Method Detection Limits (non-CLP parameters). Quantitation and detection limits are further discussed in Section 7.2.1.

1.4.3 Data Quality Objectives

Data quality objectives for the Fridley OU3 RI were developed in accordance with current U.S. EPA guidance. The DQO development process is outlined in detail in Section 4.0 of the attendant Work Plan (Volume I).

1.5 SAMPLE NETWORK DESIGN AND RATIONALE

The sample network design and rationale is discussed in detail in Section 2.0 of the attendant Field Sampling Plan (Volume II). Figures displaying the location of all proposed borings and monitoring wells are provided in Section 2.0 of the Field Sampling Plan.

1.6 PROJECT SCHEDULE

The project schedule is provided in Section 6.0 of the attendant project Work Plan (Volume I).

TABLE 1-4

ANALYTICAL DETECTION LIMITS - TCL ORGANICS
NIROP FRIDLEY, MINNESOTA
PAGE 1 OF 4

Parameter	PQL(1)	CRQL(2)	MDL(3)	
	AQ(4)	SO(5)	AQ	SO
Volatile Organic Compounds	µg/L	µg/kg	µg/L	µg/kg
Acetone	5	10	2.88	1.72
Benzene	1	10	0.076	0.14
Bromodichloromethane	1	10	0.13	0.09
Bromoform	1	10	0.15	0.13
Bromomethane	1	10	0.37	2.40
2-Butanone	5	10	0.70	1.26
Carbon disulfide	1	10	0.20	0.16
Carbon tetrachloride	1	10	0.10	0.07
Chlorobenzene	1	10	0.12	0.11
Chloroethane	1	10	0.19	2.24
Chloroform	1	10	0.17	2.08
Chloromethane	1	10	0.15	2.31
Dibromochloromethane	1	10	0.12	0.19
1,1-Dichloroethane	1	10	0.16	2.33
1,2-Dichloroethane	1	10	0.11	0.14
1,1-Dichloroethene	1	10	0.17	0.53
cis-1,2-Dichloroethene(7)	1	10	0.14	2.04
trans-1,2-Dichloroethene(7)	1	10	0.17	2.06
1,2-Dichloropropane	1	10	0.14	0.13
cis-1,3-Dichloropropene	1	10	0.15	0.19
trans-1,3-Dichloropropene	1	10	0.11	0.18
Ethylbenzene	1	10	0.11	0.10
2-Hexanone	5	10	0.50	0.85
4-Methyl-2-pentanone	5	10	0.59	0.62
Methylene chloride	2	10	1.74	7.92
Styrene	1	10	0.078	0.08
1,1,2,2-Tetrachloroethane	1	10	0.23	0.14
1,1,1-Trichloroethane	1	10	0.13	0.08
1,1,2-Trichloroethane	1	10	0.15	0.14
Trichloroethene	1	10	0.15	0.09
Tetrachloroethene	1	10	0.12	0.24
Toluene	1	10	0.13	0.18
Vinyl chloride	0.3	10	0.22	2.26
Xylenes (total)	1	10	0.13	0.16

TABLE 1-4

ANALYTICAL DETECTION LIMITS - TCL ORGANICS
NIROP FRIDLEY, MINNESOTA
PAGE 2 OF 4

Parameter	CRQL(2)		MDL(3)	
	AQ(4)	SO(5)	AQ	SO
Semivolatile Organic Compounds	µg/L	µg/kg	µg/L	µg/kg
Acenaphthene	10	330	0.24	4.9
Acenaphthylene	10	330	0.24	4.4
Anthracene	10	330	0.42	6.6
Benzo(a)anthracene	1(6)	330	0.16	7.2
Benzo(a)pyrene	1(6)	330	0.15	8.7
Benzo(b)fluoranthene	1(6)	330	0.47	13.7
Benzo(g,h,i)perylene	10	330	0.49	10.8
Benzo(k)fluoranthene	5(6)	330	0.31	8.6
Bis(2-chloroethoxy)methane	10	330	0.24	6.7
Bis(2-chloroethyl)ether	1(6)	330	0.21	3.2
Bis(2-ethylhexyl)phthalate	10	330	0.87	10.3
4-Bromophenyl-phenylether	10	330	0.27	9.9
Butylbenzylphthalate	10	330	0.12	7.7
Carbazole	10	330	0.24	6.1
4-Chloro-3-methylphenol	10	330	0.33	11.1
4-Chloroaniline	10	330	1.06	38
2-Chloronaphthalene	10	330	0.44	8.4
2-Chlorophenol	10	330	0.19	5.6
4-Chlorophenyl-phenylether	10	330	0.35	6.6
Chrysene	10	330	0.19	5.1
Dibenz(a,h)anthracene	1(6)	330	0.12	7.6
Dibenzofuran	10	330	0.27	5.5
3,3'-Dichlorobenzidine	10	330	1.93	79.7
Diethylphthalate	10	330	0.17	8.2
Di-n-butylphthalate	10	330	0.18	7.0
Di-n-octylphthalate	10	330	0.40	6.7
4,6-Dinitro-2-methylphenol	25	830	1.19	116
2,4-Dinitrophenol	10(6)	830	1.08	15
2,4-Dinitrotoluene	2(6)	330	0.22	12.4
1,2-Dichlorobenzene	10	330	0.51	5.4
1,3-Dichlorobenzene	10	330	0.60	3.2
1,4-Dichlorobenzene	10	330	0.51	3.6
2,4-Dichlorophenol	10	330	0.26	10.5

TABLE 1-4

ANALYTICAL DETECTION LIMITS - TCL ORGANICS
NIROP FRIDLEY, MINNESOTA
PAGE 3 OF 4

Parameter	CRQL(2)		MDL(3)	
	AQ(4)	SO(5)	AQ	SO
Semivolatile Organic Compounds	µg/L	µg/kg	µg/L	µg/kg
Dimethylphthalate	10	330	0.21	12.2
2,4-Dimethylphenol	10	330	1.18	67
2,6-Dinitrotoluene	2(6)	330	0.29	27.0
Fluoranthene	10	330	0.41	5.6
Fluorene	10	330	0.16	4.4
Hexachlorobenzene	1(6)	330	0.26	9.5
Hexachlorobutadiene	1(6)	330	0.54	4.0
Hexachlorocyclopentadiene	10	330	1.38	6.7
Hexachloroethane	2(6)	330	0.57	5.2
Indeno(1,2,3-cd)pyrene	1(6)	330	0.06	7.0
Isophorone	10	330	0.18	8.2
2-Methylnaphthalene	10	330	0.41	6.4
2-Methylphenol	3(6)	330	0.69	9.4
4-Methylphenol	10	330	0.43	8.6
Naphthalene	10	330	0.38	5.7
2-Nitroaniline	25	830	0.26	10.3
3-Nitroaniline	25	830	1.16	89.3
4-Nitroaniline	25	830	3.14	81
Nitrobenzene	10	330	0.55	9.2
2-Nitrophenol	10	330	0.34	10.9
4-Nitrophenol	25	830	1.74	4.2
N-nitroso-di-n-propylamine	10	330	0.30	8.0
N-nitrosodiphenylamine	10	330	0.29	12.5
2,2'-Oxybis(1-chloropropane)	10	330	0.26	9.8
Pentachlorophenol	10(6)	830	1.2	6.3
Phenanthrene	10	330	0.24	6.4
Phenol	10	330	0.28	30.1
Pyrene	10	330	0.20	6.8
1,2,4-Trichlorobenzene	10	330	0.54	8.1
2,4,5-Trichlorophenol	25	830	0.28	9.3
2,4,6-Trichlorophenol	10	330	0.15	4.8
Pyridine(8)	10	330	0.65	83

TABLE 1-4

ANALYTICAL DETECTION LIMITS - TCL ORGANICS
NIROP FRIDLEY, MINNESOTA
PAGE 4 OF 4

Parameter	CRQL(2)		MDL(3)	
	AQ(4)	SO(5)	AQ	SO
Polychlorinated biphenyls	µg/L	µg/kg	µg/L	µg/kg
Aroclor-1016	0.5(6)	33	0.081	5.36
Aroclor-1221	1.0(6)	67	0.092	9.58
Aroclor-1232	0.5(6)	33	0.17	4.34
Aroclor-1242	0.5(6)	33	0.3	6.65
Aroclor-1248	0.5(6)	33	0.091	18
Aroclor-1254	0.5(6)	33	0.1	22
Aroclor-1260	0.5(6)	33	0.084	3.36

1 PQL Practical Quantitation Limit.
2 CRQL Contract Required Quantitation Limit; as specified in OLM03.1, unless otherwise noted.
3 MDL Method Detection Limit; as provided by Laucks Testing Laboratories, Inc.
4 AQ Aqueous (groundwater) samples.
5 SO Solid (soil) samples.
6 CRQL revised to reflect laboratory's "true" reporting limit since standard CRQL for this compound exceeds MPCA HRL or other MPCA groundwater criterion.
7 1,2-Dichloroethene is typically reported as total 1,2-dichloroethene based on CLP requirements. The cis- and trans-isomers of 1,2-dichloroethene will be individually reported for the OU3 RI.
8 Pyridine is not part of the CLP TCL list but will be included in the semivolatile analysis of the OU3 RI samples since this compound was a component of products used at the NIROP Fridley.

TABLE 1-5

ANALYTICAL DETECTION LIMITS - TAL INORGANICS
NIROP FRIDLEY, MINNESOTA

Parameter	CRDL(1)		IDL(2)	
	AQ(3)	SO(4)	AQ	SO
Target Analyte List Metals	µg/L	mg/kg	µg/L	mg/kg
Aluminum	200	40	68	13.6
Antimony	60	12	12	2.4
Arsenic	10	2	1.9	0.38
Barium	200	40	0.5	0.1
Beryllium	5	1	0.3	0.06
Cadmium	5	1	3	0.6
Calcium	5000	1000	54	10.8
Chromium	10	2	5	1
Cobalt	50	10	2	0.4
Copper	25	5	2	0.4
Cyanide	10	10	2.686(5)	0.0238(5)
Iron	100	20	22	4.4
Lead	3	0.6	0.79	0.16
Magnesium	5000	1000	55	11
Manganese	15	3	1	0.2
Mercury	0.2	0.1	0.025	0.025
Nickel	40	8	3	0.6
Potassium	5000	1000	96	19.2
Selenium	5	1	1	0.2
Silver	10	2	3	0.6
Sodium	5000	1000	20	4
Thallium	10	2	0.78	0.16
Vanadium	50	10	3	0.6
Zinc	20	4	2	0.4

1 CRDL Contract Required Detection Limit; as specified in ILM04.0.
2 IDL Instrument Detection Limit, unless otherwise noted; as provided by Laucks Testing Laboratories, Inc.
3 AQ Aqueous (groundwater) samples.
4 SO Solid (soil) samples.
5 MDL Method Detection Limit; as specified by Laucks Testing Laboratories, Inc.

TABLE 1-6

ANALYTICAL DETECTION LIMITS
BIOLOGICAL/ENGINEERING/MISCELLANEOUS PARAMETERS
NIROP FRIDLEY, MINNESOTA

Parameter	Aqueous Samples (mg/L)	
	PQL[1]	MDL[2]
Total Suspended Solids	2	NA[3]
Alkalinity (as $CaCO_3$)	2	NA
Hardness (as $CaCO_3$)	1	NA
Sulfate	1	0.057
Nitrate	0.2	0.01
Nitrite	0.1	0.025
Dissolved Chloride	1	0.1
Dissolved Bromide	1	0.012
Dissolved Phosphate	1	0.12
Dissolved Methane (ng/L)	15	5.03
	Solid Samples (mg/Kg)[4]	
Total Organic Carbon	200	24
Hexavalent Chromium	2	0.6
pH (pH units)	±0.1[5]	NA
Ferrous Iron	estimated at 1[5]	NA
Sulfide	estimated at 40[5]	NA
Oxidation Reduction Potential (mV)	±10 mV[5]	NA

1 PQL Practical Quantitation Limit; as provided by Laucks Testing Laboratories, Inc.
2 MDL Method Detection limit; as provided by Laucks Testing Laboratories, Inc.
3 NA MDL determination not applicable to this method.
4 Units for solid sample results are mg/kg unless otherwise noted.
5 PQL not applicable. Values shown represent sensitivity for the parameter.

2.0 PROJECT ORGANIZATION

The project organization for the OU3 Remedial Investigation/Feasibility Study is provided in Section 7.0 (Project Management) of the attendant Work Plan (Volume I). A project organization chart, management responsibilities, quality assurance responsibilities, laboratory responsibilities, and field responsibilities are discussed in Sections 7.1 through 7.5 of the Work Plan, respectively.

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 results which are legally defensible in a court of law. 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, reporting of data, internal quality control, 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 and/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 for this project is described in detail 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 solid matrix samples are considered to be precise if the relative percent difference (RPD) is less than or equal to 50 percent. Field duplicate results for aqueous matrix samples are considered to be precise if the 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 analytical samples.

3.1.3 Field GC Precision Objectives

Precision for field GC analyses will be measured through the use of field duplicates and laboratory duplicates. Field duplicates, as specified in Section 3.1.2, will be collected at a rate of one duplicate per ten environmental samples. Laboratory duplicate analysis for field GC analyses will be performed by analyzing two aliquots of the same sample at a frequency of one duplicate per 20 environmental samples. Sampling personnel will identify samples to be used for laboratory duplicate analysis on the chain-of-custody report (COC) and will supply extra volume for such samples. If any of the three largest component peaks for the target compounds listed in Table 1-3 in the field or laboratory duplicate sample are above the PQL in both analyses but exhibit a Relative Percent Difference (RPD) exceeding 150%, or if any site-specific target compound is detected in one analysis at a level greater than 5-times the PQL but is not detected in the duplicate analysis, the Field Operations Leader (FOL) shall be informed and a third aliquot or a fresh sample obtained from the same location shall be analyzed. Further detail regarding laboratory and field duplicate analysis for the field GC is provided in Sections 5.9.5 and 5.9.6, respectively, in the field GC SOP (Appendix C).

3.1.4 Laboratory Precision Objectives

Laboratory precision Quality Control samples will be analyzed with a frequency of 5 percent (i.e., one quality control sample per 20 environmental samples) for organic analyses and a frequency of 10 percent (i.e., one quality control sample per 10 environmental samples) for inorganic analyses. Laboratory precision is measured via comparison of calculated Relative Percent Difference (RPD) values and Precision Control Limits specified in the analytical method or by the laboratory's QA/QC Program.

Five distinct types of analyses will be completed for environmental samples collected during the OU3 RI at the NIROP Fridley, as follows (Laucks Testing Laboratories, Inc., SOPs are provided in Appendix A):

- Target Compound List (TCL) organic analyses via OLM03.1 and SOP LTL-8260A. As discussed in Section 1.4.2.2, analysis for volatiles in aqueous samples only will be performed via SOP LTL-8260A with a 25 mL sample volume The remaining TCL organic analyses will be performed via OLM03.1. Analysis for PCBs will be modified to focus on PCB-only analyses as described in the Addendum to Laucks SOP LTL-8082 (also provided in Appendix A).

- Target Analyte List (TAL) inorganic/cyanide analysis via ILM04.0.

- Total Organic Carbon (TOC), hexavalent chromium, pH, ORP, sulfide, and ferrous iron analyses of soil via SOPs LTL-7014, LTL-9113, LTL-9128, LTL-9301, and LTL-7601.

- Characteristic analyses to evaluate natural attenuation of chlorinated solvents including sulfate, nitrate, nitrite, dissolved chloride, dissolved bromide, dissolved phosphate, and dissolved methane via SOPs LTL-9110 and AM18.

- General water quality analyses including total suspended solids, hardness (as $CaCO_3$), and alkalinity (as $CaCO_3$) via SOPs LTL-9202, LTL-9009, and LTL-9005.

Precision for TCL organic analysis will be measured via the RPDs for matrix spike/matrix spike duplicate samples. Precision for TAL inorganic analysis will be measured via RPDs for laboratory duplicates. Precision for dissolved methane will be measured via the RPD for field duplicates. Precision for the remaining parameters will be measured via the RPD results for laboratory duplicate samples. Tables 3-1 through 3-3 present RPD Precision Control Limits.

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 for this project is described in detail 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 times. Accuracy and precision requirements for field measurements (e.g., pH) are ensured through calibration as discussed in Section 9.1 of the Field Sampling Plan.

3.2.3 Field GC Accuracy Objectives

Accuracy for field GC analyses will be measured through the use of matrix spikes, QC check standards, and blanks. Matrix spike analyses will be performed at a frequency of 5% (one matrix spike per twenty

TABLE 3-1

PRECISION CONTROL LIMITS (RPDS)[1]
MATRIX SPIKE/MATRIX SPIKE DUPLICATE SAMPLES
ORGANIC ANALYSIS VIA OLM03.1[2,3]
NIROP FRIDLEY, MINNESOTA

Chemical	Aqueous Samples	Solid Samples
VOLATILE ORGANICS		
1,1-Dichloroethene	20	22
Trichloroethene	20	24
Benzene	20	21
Toluene	20	21
Chlorobenzene	20	21
SEMIVOLATILE ORGANICS		
Phenol	42	35
2-Chlorophenol	40	50
1,4-Dichlorobenzene	28	27
N-Nitroso-di-n-propylamine	38	38
1,2,4-Trichlorobenzene	28	23
4-Chloro-3-methylphenol	42	33
Acenaphthene	31	19
4-Nitrophenol	50	50
2,4-Dinitrotoluene	38	47
Pentachlorophenol	50	47
Pyrene	31	36
PCBs		
Aroclor 1016	35	50
Aroclor 1260	35	50

1 RPD - Relative Percent Difference as described in Section 12.0.
2 U.S. EPA (U.S. Environmental Protection Agency) CLP, 1994. Statement of Work for Organics Analysis, Multi-Media, Multi-Concentration, OLM03.1.
3 As noted previously, volatile analysis for aqueous samples will be performed using SOP LTL-8260 (Appendix A).

TABLE 3-2

PRECISION CONTROL LIMITS (RPDS)(1)
LABORATORY DUPLICATE SAMPLES
INORGANIC ANALYSIS VIA ILM04.0(2)
NIROP FRIDLEY, MINNESOTA

Chemical	Aqueous Samples	Solid Samples
INORGANICS		
Aluminum	20	35
Antimony	20	35
Arsenic	20	35
Barium	20	35
Beryllium	20	35
Cadmium	20	35
Calcium	20	35
Chromium	20	35
Cobalt	20	35
Copper	20	35
Iron	20	35
Lead	20	35
Magnesium	20	35
Manganese	20	35
Mercury	20	35
Nickel	20	35
Potassium	20	35
Selenium	20	35
Silver	20	35
Sodium	20	35
Thallium	20	35
Vanadium	20	35
Zinc	20	35
Cyanide	20	35

1 RPD - Relative Percent Difference as described in Section 12.0.
2 U.S. EPA (U.S. Environmental Protection Agency) CLP, 1995. Statement of Work for Inorganics Analysis, Multi-Media, Multi-Concentration, ILM04.0.

TABLE 3-3

PRECISION CONTROL LIMITS (RPDS)(1)
BIOLOGICAL/ENGINEERING/MISCELLANEOUS PARAMETERS
LABORATORY DUPLICATE SAMPLES
NIROP FRIDLEY, MINNESOTA

Parameter	Aqueous Samples
Total Suspended Solids	20
Alkalinity(as $CaCO_3$)	10
Hardness (as $CaCO_3$)	15
Sulfate	10
Nitrate	10
Nitrite	30
Dissolved Chloride	11
Dissolved Bromide	30(3)
Dissolved Phosphate	30(3)
Dissolved Methane	NA(2)
	Solid Samples
Total Organic Carbon	33
Hexavalent Chromium	20(4)
pH	±0.5 pH units
Ferrous Iron	qualitative confirmation
Sulfide	qualitative confirmation
Oxidation-Reduction Potential	20(3)

1 RPD - Relative Percent Difference as described in Section 12.0.
2 Not Applicable.
3 Default limits; insufficient data points available to generate statistical laboratory control limits.
4 Default limits specified by SW-846 Method 3060A.

environmental samples). Sampling personnel will identify samples to be used for matrix spike analysis on the COC and will supply extra volume for such samples. Matrix spike samples will be spiked with each of the target compounds shown in Table 1-3. Accuracy control limits of 50 to 150 percent will be used to assess matrix spike recovery for target compounds. Further information regarding matrix spikes for field GC analysis is provided in Section 5.9.4 of the field GC SOP (Appendix C).

A QC check standard solution containing all target compounds listed in Table 1-3 will be analyzed with each initial calibration. Accuracy control limits for QC check standard Percent Recoveries (%Rs) will be 50 to 150 percent. Analysis of the QC check standard solution is further discussed in Section 5.9.1 of field GC SOP (Appendix C).

Equipment rinsate blanks (one per ten environmental samples, with a minimum of one per day), trip blanks (one per cooler), and method or laboratory reagent blanks (after each initial and continuing calibration) will also be analyzed to assess accuracy. These types of blanks are described in more detail in Section 3.6 of this QAPP. Further detail regarding control limits and corrective actions for these blanks for PGC analysis is provided Sections 5.9.2 and 5.9.3 of the field GC SOP (Appendix C).

Retention time monitoring and control, as fully described in Section 5.9.7 of the field GC SOP (Appendix C), will also be performed to monitor the accuracy of qualitative analyte identification.

3.2.4 Laboratory Accuracy Objectives

Accuracy in the laboratory is measured through the comparison of a spiked sample result against a known or calculated value expressed as a percent recovery (%R). Percent recoveries are derived from the analysis of known amounts of compounds spiked into deionized water (i.e., laboratory control sample analysis), or into actual samples (i.e., surrogate or matrix spike analysis). Laboratory control sample analysis measures the accuracy of laboratory operations. Surrogate and matrix spike analyses measure the accuracy of laboratory operations as affected by matrix. Laboratory control sample analyses are performed with a frequency of one per twenty associated samples of like matrix. Matrix spike analyses will be performed with a frequency of one per twenty associated samples of like matrix for organic analyses and with a frequency of one per ten associated samples of like matrix for inorganic analyses. Surrogate spike analysis is performed for all organic chromatographic analyses. Laboratory accuracy is assessed via comparison of calculated percent recovery (%R) values with Accuracy Control Limits specified in the analytical method or by the laboratory's QA/QC Program.

Five distinct types of analyses will be completed for environmental samples collected during the OU3 RI at the NIROP Fridley, as follows (Laucks Testing Laboratories, Inc., SOPs are provided in Appendix A):

- Target Compound List (TCL) organic analyses via OLM03.1 and SOP LTL-8260A. As discussed in Section 1.4.2.2, analysis for volatiles in aqueous samples only will be performed via SOP LTL-8260A with a 25 mL sample volume The remaining TCL organic analyses will be performed via OLM03.1. Analysis for PCBs will be modified to focus on PCB-only analyses as described in the Addendum to Laucks SOP LTL-8082 (also provided in Appendix A).

- Target Analyte List (TAL) inorganic/cyanide analysis via ILM04.0.

- Total Organic Carbon (TOC), hexavalent chromium, pH, ORP, sulfide, and ferrous iron analyses of soil via SOPs LTL-7014, LTL-9113, LTL-9128, LTL-9301, and LTL-7601.

- Characteristic analyses to evaluate natural attenuation of chlorinated solvents including sulfate, nitrate, nitrite, dissolved chloride, dissolved bromide, dissolved phosphate, and dissolved methane via SOPs LTL-9110 and AM18.

- General water quality analyses including total suspended solids, hardness (as $CaCO_3$), and alkalinity (as $CaCO_3$) via SOPs LTL-9202, LTL-9009, and LTL-9005.

Accuracy for Target Compound List organic analysis will be measured via the percent recoveries for surrogate spikes and matrix spike/matrix spike duplicates. Accuracy for Target Analyte List Inorganic analysis will be measured via percent recoveries for matrix spikes and laboratory control samples. Accuracy the remaining analytes will be measured via percent recoveries for matrix spikes and laboratory control samples, as applicable. Tables 3-4 and 3-5 present control limits for matrix and surrogate spike recoveries, respectively, for TCL organics. Tables 3-6 and 3-7 present control limits for matrix spike and laboratory control samples, respectively, for TAL inorganics. Tables 3-8 and 3-9 present control limits for matrix spikes and laboratory control samples, respectively, for the remaining, non-CLP parameters.

TABLE 3-4

ACCURACY CONTROL LIMITS (%R)(1)
MATRIX SPIKE/MATRIX SPIKE DUPLICATE SAMPLES
ORGANIC ANALYSIS VIA OLM03.1(2,3)
NIROP FRIDLEY, MINNESOTA

Chemical	Aqueous Samples	Solid Samples
VOLATILE ORGANICS		
1,1-Dichloroethene	60-140	59-172
Trichloroethene	60-140	62-137
Benzene	60-140	66-142
Toluene	60-140	59-139
Chlorobenzene	60-140	60-133
SEMIVOLATILE ORGANICS		
Phenol	12-110	26-90
2-Chlorophenol	27-123	25-102
1,4-Dichlorobenzene	36-97	28-104
N-Nitroso-di-n-propylamine	41-116	41-126
1,2,4-Trichlorobenzene	39-98	38-107
4-Chloro-3-methylphenol	23-97	26-103
Acenaphthene	46-118	31-137
4-Nitrophenol	10-80	11-114
2,4-Dinitrotoluene	24-96	28-89
Pentachlorophenol	9-103	17-109
Pyrene	26-127	35-142
PCBs		
Aroclor 1016	40-160	40-160
Aroclor 1260	39-149	40-160

1 %R - Percent Recovery as described in Section 12.0.
2 U.S. EPA (U.S. Environmental Protection Agency) CLP, 1994. Statement of Work for Organics Analysis, Multi-Media, Multi-Concentration, OLM03.1.
3 As noted previously, volatile analysis for aqueous samples will be performed using SOP LTL-8260 (Appendix A).

TABLE 3-5

ACCURACY CONTROL LIMITS (%R)(1)
SURROGATE SPIKES
ORGANIC ANALYSIS VIA OLM03.1(2,3)
NIROP FRIDLEY, MINNESOTA

Chemical	Aqueous Samples	Solid Samples
VOLATILE ORGANICS		
Toluene-d8	60-140	84-138
Bromoflourobenzene	60-140	59-113
1,2-Dichloroethane-d4	60-140	70-121
SEMIVOLATILE ORGANICS		
Nitrobenzene-d5	35-114	23-120
2-Fluorobiphenyl	43-116	30-115
Terphenyl-d14	33-141	18-137
Phenol-d5	10-110	24-113
2-Fluorophenol	21-110	25-121
2,4,6-Tribromophenol	10-123	19-122
2-Chlorophenol-d4	33-110(4)	20-130(4)
1,2-Dichlorobenzene-d4	16-110(4)	20-130(4)
PCBs		
Tetrachloro-m-xylene	30-150	30-150
Decachlorobiphenyl	30-150	30-150

1 %R - Percent Recovery as described in Section 12.0.
2 U.S. EPA (U.S. Environmental Protection Agency) CLP, 1994. Statement of Work for Organics Analysis, Multi-Media, Multi-Concentration, OLM03.1.
3 As noted previously, volatile analysis for aqueous samples will be performed using SOP LTL-8260 (Appendix A).
4 Advisory limits only.

TABLE 3-6

ACCURACY CONTROL LIMITS (%R)(1)
MATRIX SPIKE SAMPLES
INORGANIC ANALYSIS VIA ILM04.0(2)
NIROP FRIDLEY, MINNESOTA

Chemical	Aqueous Samples	Solid Samples
INORGANICS		
Aluminum	75-125	NS(3)
Antimony	75-125	75-125
Arsenic	75-125	75-125
Barium	75-125	75-125
Beryllium	75-125	75-125
Cadmium	75-125	75-125
Calcium	NS(3)	NS(3)
Chromium	75-125	75-125
Cobalt	75-125	75-125
Copper	75-125	75-125
Iron	75-125	75-125
Lead	75-125	75-125
Magnesium	NS(3)	NS(3)
Manganese	75-125	75-125
Mercury	75-125	75-125
Nickel	75-125	75-125
Potassium	NS(3)	NS(3)
Selenium	75-125	75-125
Silver	75-125	75-125
Sodium	NS(3)	NS(3)
Thallium	75-125	75-125
Vanadium	75-125	75-125
Zinc	75-125	75-125
Cyanide	75-125	75-125

1 %R - Percent Recovery as described in Section 12.0.
2 U.S. EPA (U.S. Environmental Protection Agency) CLP, 1995. Statement of work for Inorganics Analysis, Multi-Media, Multi-Concentration, ILM04.0.
3 No spike required.

TABLE 3-7

ACCURACY CONTROL LIMITS (%R)(1)
LABORATORY CONTROL SAMPLES
INORGANIC ANALYSIS VIA ILM04.0(2)
NIROP FRIDLEY, MINNESOTA

Chemical	Aqueous Samples	Solid Samples
INORGANICS		
Aluminum	80-120	TBD(3)
Antimony	80-120(4)	TBD
Arsenic	80-120	TBD
Barium	80-120	TBD
Beryllium	80-120	TBD
Cadmium	80-120	TBD
Calcium	80-120	TBD
Chromium	80-120	TBD
Cobalt	80-120	TBD
Copper	80-120	TBD
Iron	80-120	TBD
Lead	80-120	TBD
Magnesium	80-120	TBD
Manganese	80-120	TBD
Mercury	80-120(5)	TBD
Nickel	80-120	TBD
Potassium	80-120	TBD
Selenium	80-120	TBD
Silver	80-120(4)	TBD
Sodium	80-120	TBD
Thallium	80-120	TBD
Vanadium	80-120	TBD
Zinc	80-120	TBD
Cyanide	NA(6)	TBD

1 %R - Percent Recovery as described in Section 12.0.
2 U.S. EPA (U.S. Environmental Protection Agency) CLP, 1995. Statement of Work for Inorganic Analysis, Multi-Media, Multi-Concentration, ILM04.0.
3 TBD - To Be Determined at time of analysis based on EPA LCS lot number.
4 Advisory limits only.
5 LCS analysis for mercury is not required by CLP protocol, but will be performed for this project.
6 NA - Not Applicable.

TABLE 3-8

ACCURACY CONTROL LIMITS (%R)(1)
BIOLOGICAL/ENGINEERING/MISCELLANEOUS PARAMETERS
MATRIX SPIKE SAMPLES
NIROP FRIDLEY, MINNESOTA

Analytical Method	Aqueous Samples
Total Suspended Solids	NA(2)
Alkalinity(as $CaCO_3$)	NA
Hardness (as $CaCO_3$)	NA
Sulfate	81-115
Nitrate	79-117
Nitrite	50-150
Dissolved Chloride	73-121
Dissolved Bromide	50-150(3)
Dissolved Phosphate	50-150(3)
Dissolved Methane	NA
	Solid Samples
Total Organic Carbon	63-119
Hexavalent Chromium	75-125(4)
pH	NA
Ferrous Iron	NA
Sulfide	NA
Oxidation-Reduction Potential	NA

1 %R - Percent Recovery as described in Section 12.0.
2 NA - Not Applicable.
3 Default limits; insufficient data points available to generate statistical laboratory control limits.
4 Default limits specified by SW-846 Method 3060A.

TABLE 3-9

ACCURACY CONTROL LIMITS (%R)(1)
BIOLOGICAL/ENGINEERING/MISCELLANEOUS PARAMETERS
LABORATORY CONTROL SAMPLES
NIROP FRIDLEY, MINNESOTA

Analytical Method	Aqueous Samples
Total Suspended Solids	NA(2)
Alkalinity(as $CaCO_3$)	88-112
Hardness (as $CaCO_3$)	87-115
Sulfate	90-110
Nitrate	90-110
Nitrite	90-110
Dissolved Chloride	90-110
Dissolved Bromide	90-110
Dissolved Phosphate	90-110
Dissolved Methane	NA
	Solid Samples
Total Organic Carbon	80-120
Hexavalent Chromium	NA
pH	NA
Ferrous Iron	NA
Sulfide	NA
Oxidation-Reduction Potential	NA

1 %R - Percent Recovery as described in Section 12.0.
2 NA - Not Applicable.

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 the OU3 RI as a whole since it is anticipated that all samples will be collected within a four-month period. If critical data points are lost, resampling and/or reanalysis may be required.

One hundred percent of the fixed-base laboratory data for the OU3 RI will be validated in accordance with the Region 5 Standard Operating Procedures for Validation of CLP Organic and Inorganic Data and the U.S. EPA Contract Laboratory Program National Functional Guidelines for Organic and Inorganic Data Review. Data rejected as a result of the validation process will be treated as incomplete 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 90 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 greater than 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 existing 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 upon the proper design of the sampling program and will be satisfied by ensuring that the Field Sampling Plan (FSP) is followed and that proper sampling techniques are used.

3.4.3 Measures to Ensure Representativeness of Lab 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 OU3 RI was designed to provide data representative of facility conditions. During development of this network, consideration was given to past waste disposal practices, existing analytical data, physical setting and processes, and constraints inherent to the CERCLA program. The rationale of the sampling network is discussed in detail in the Field Sampling Plan (FSP).

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 and reporting of solid matrix sample results on a dry-weight basis). Additionally, consideration is given to seasonal conditions and other environmental variations that could exist to influence data results.

3.5.2 Measures to Ensure Comparability of Field Data

Comparability is dependent upon the proper design of the sampling program and will be satisfied by ensuring that the FSP is followed and that proper sampling techniques are used. It is also dependent on recording field measurements using the correct units. Field measurements for this project include pH, specific conductance, temperature, turbidity, dissolved oxygen, dissolved ferrous iron, dissolved reduced manganese, hydrogen sulfide, oxidation-reduction potential, and volatile organic compounds by field GC analysis. The units used for the field measurements for this project are as follows:

- pH is measured to the nearest 0.1 standard pH unit.
- Specific conductance is measured in millimhos (the inverse of the ohm).
- Temperature is measured in degrees Celsius.
- Turbidity is measured in nephelometric turbidity units (NTU).
- Dissolved oxygen is measured in mg/L.
- Dissolved ferrous iron is measured in mg/L.
- Dissolved reduced manganese is measured in mg/L.
- Hydrogen sulfide is measured in mg/L.
- Oxidation Reduction Potential is measured in mV.
- Volatile organics by field GC are measured in µg/kg.

3.5.3 Measures to Ensure Comparability of Lab Data

Planned analytical data will be comparable when similar sampling and analytical methods are used and documented. Results will be reported in units that ensure comparability with previous data and with current state and Federal standards and guidelines. Organic chemicals will be reported in µg/L for aqueous samples and µg/kg for solid samples. Metals and cyanide will be reported as µg/L for aqueous samples and mg/kg for solid samples. Total organic carbon and hexavalent chromium will be reported in mg/kg (solid samples). Oxidation-reduction potential and pH in soils will be reported in standard pH units and mV, respectively. Ferrous iron and sulfide will be reported as qualitatively present or absent in soils. The remaining biological/engineering parameters will be reported in mg/L (aqueous samples). Detection/reporting limits are further discussed in Sections 7.2.1 and 1.4.2.2

3.6 LEVEL OF QUALITY CONTROL EFFORT

Trip blank, rinsate blank, ambient condition blank, source water blank, method blank, duplicate, standard reference materials (SRM) and matrix spike samples will be analyzed to assess the quality of the data resulting from the field sampling and analytical programs. In addition, duplicate measurements will be completed for field parameters.

External QC measures (i.e., field quality control samples) consist of field duplicates, ambient condition blanks, trip blanks, source water 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 quality control samples undergo the same preservation, analysis, and reporting procedures as the related environmental samples. Each type of field quality control sample is discussed below.

Field duplicates are either two samples collected independently at a sampling location (e.g., surface water), or a single sample homogenized and split into two portions (where volatile organic compounds (VOCs) are to be analyzed, the VOC sample aliquots are containerized first to avoid loss of constituents, then the remaining sample matrix is homogenized.) Field duplicates are collected and analyzed for chemical constituents to measure the precision of the sampling and analysis methods employed. The general level of the QC effort will be one field duplicate for every 10 or fewer investigative samples.

Trip blanks and ambient condition blanks, consisting of distilled water, will be submitted to the Laucks Testing Laboratories, Inc., to provide the means to assess the quality of the data resulting from the field sampling program. Ambient blank samples are analyzed to check for interfering contaminants that could potentially be present in ambient air at the sampling site (e.g., volatile compounds or particulates). Ambient blanks will be collected based on conditions at the time of sampling at the discretion of the Field Operations Leader (FOL), with a minimum of one ambient blank being collected during the RI. Trip blanks pertain to volatile organic compounds (VOCs) only. Trip blanks are used to assess the potential for contamination of VOCs resulting from contaminant migration into sample bottles/jars during sample shipment and storage. Trip blanks are prepared by the laboratory prior to the sampling event, 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 for analysis. There should be one trip blank included in each sample shipping container that contains VOCs. At no time after trip blank preparation are their sample containers opened before they reach the laboratory.

Equipment rinsate blanks 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 will be collected per each type of sampling equipment used (i.e., bailer, split-spoon sampler, hand tools, etc.) per day that sampling is conducted. A sampling event is matrix specific, therefore an equipment blank must be collected for each matrix sampled. 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 consist of potable waters used in decontamination and steam cleaning activities. Source water blanks are analyzed for all organic and inorganic constituents under investigation as a means of determining whether the source waters used in decontamination activities have introduced contaminants to the environmental samples. Source water blanks will be collected at a rate of one per each potable water source.

Method blank samples are generated within the laboratory and used to assess contamination resulting from laboratory procedures. Laboratory duplicate samples are analyzed for inorganic parameters to check for sampling and analytical reproducibility. Matrix spikes provide information about the effect of the sample matrix on the digestion and measurement methodology. Laboratory duplicates and matrix spikes for inorganic analyses will be analyzed with a frequency of ten percent (one per every ten or fewer investigative samples per matrix (i.e., groundwater, soil). All matrix spikes for organic analyses are performed in duplicate and are hereinafter referred to as MS/MSD samples.

MS/MSD samples are investigative samples. Soil MS/MSD samples require no extra volume for VOCs or extractable organics. However, extra sample volume must be collected for aqueous MS/MSD samples for VOCs and extractable organics. Specifically, 4 extra 40 mL bottles for VOCs, 2 extra 1000 mL bottles for semivolatiles, and 2 extra 1000 mL bottles for PCBs are required. One MS/MSD sample will be collected/designated for every 20 or fewer investigative samples per sample matrix (i.e. groundwater, soil) for organic analyses.

The level of QC effort for testing of Target Compound List (TCL) organics (volatiles in soil samples and semivolatiles) will conform to the Statement of Work (SOW/OLM03.1). Modifications for PCB-only analysis are provided in the Addendum to Laucks SOP LTL-8082 (Appendix A). The level of QC effort for TCL volatiles in aqueous samples will conform to SOP LTL-8260 (Appendix A). The level of QC effort for

all inorganic parameters will exceed method requirements in that matrix spike and laboratory duplicate analyses will be performed after every 10 investigative samples instead of after every 20 investigative samples. With this exception, the level of QC effort for testing of inorganics (metals and cyanide) will conform to the Statement of Work (SOW/ILM04.0) and the level of QC effort for testing of all non-CLP analytes will conform to the SOPs provided in Appendix A.

4.0 SAMPLING PROCEDURES

Field sampling procedures for the Operable Unit 3 Remedial Investigation are discussed in detail in the attendant Field Sampling Plan (Volume II). The specific sampling information components required by U.S. EPA Region V as outlined in the CERCLA model Quality Assurance Project Plan and their location in the Field Sampling Plan (FSP) are as follows:

- Field sampling by matrix - Section 2.0 of the FSP
- Field quality control sample collection/preparation procedures - Section 8.0 of the FSP
- Sample containers, preservatives, and volume requirements - Section 4.0 of the FSP
- Decontamination procedures - Section 6.0 of the FSP
- Sample packaging and shipping procedures - Section 5.0 of the FSP

In addition, Sections 7 through 11 of the Field Sampling Plan address the following sampling procedures and field investigation tasks:

- Mobilization/demobilization - Section 7.1
- Monitoring well installation - Section 7.4
- Monitoring well development - Section 7.4.1
- Groundwater sampling - Section 7.5
- Water level measurements - Section 7.4.2
- Soil sampling procedures - Section 7.3
- Surveying - Section 7.6
- Aquifer testing - Section 7.7
- Waste handling - Section 7.8
- Quality control sample procedures - Section 8.0
- Field measurements/screening - Section 9.0
- Preventive maintenance procedures/schedule - Section 10.0
- Sample disposal - Section 11.0

Standard Operating Procedures regarding sampling and record keeping are included as Appendices to the Field Sampling Plan.

5.0 CUSTODY PROCEDURES

Custody is one of several factors which is 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 and purge files, 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, airbill number, laboratory sample custodian), the COC report documents sample custody and tracking. Custody procedures apply to all environmental and associated field quality control samples obtained as part of the data collection system.

5.1 FIELD CUSTODY PROCEDURES

The FOL (or designee) is responsible for the care and custody of the samples collected until they are relinquished to the 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 will be affixed to the sample bottles and will be returned by the analytical laboratory for inclusion in the final evidence file. COC report forms and sample labels are generally supplied by the laboratory subcontractor. In accordance with

NFESC guidelines, samples for chemical constituents analysis must be sent (for next-day receipt) to the laboratory within 24-hours of collection.

The field GC will be located in a building at the NIROP Fridley, typically within five minutes driving time from all sample collection locations. Samples, along with completed COC reports, will be hand-delivered by field personnel to the GC analyst. At times, the analyst may also pick up samples from the collection sites. The Field Operations Leader is responsible for maintaining COC procedures until the time of sample delivery or pickup. After that time, the analyst is responsible for maintaining COC procedures and for refrigeration of all samples until all analyses have been successfully completed.

Full details regarding sample chain-of-custody (including use of custody seals and sample shipment protocols) are contained in B&R Environmental SOP SA-6.1, which is provided in as an appendix to the attendant Field Sampling Plan (Volume II). B&R Environmental SOP SA-6.2, also provided in the FSP, discusses maintenance of site logbooks, site notebooks, and other field records. Additionally, each of the various sampling SOPs incorporated into the FSP contains a section that addresses relevant sample documentation (i.e., completion of sample logsheets, etc.). All sample records are eventually docketed into the B&R Environmental project central file.

5.2 LABORATORY CUSTODY PROCEDURES

When samples are received by the laboratory subcontractor, the laboratory's sample custodian will examine each cooler's custody seals to verify that they are intact and that the integrity of the environmental samples has been maintained. The custodian will then open the cooler and measure its internal temperature. The temperature reading will be noted on the Supplemental Sample Receipt Log, as further discussed below. The sample custodian will then sign the COC report and examine the contents of the cooler. Sample container breakages or discrepancies between the COC report and sample label documentation will be recorded. With the exception of samples for volatiles analysis, the pH of chemically preserved samples will be checked using Hydrion paper and recorded. (The pH of volatile samples will be checked and recorded after analysis to prevent loss of volatile compounds.) A Laucks Testing Laboratories, Inc., CLP Sample Receipt Log and Supplemental Sample Receipt Log, as shown in Appendix 3 of LTL 4002 (Appendix A), are also completed. All problems or discrepancies noted during this process are to be promptly reported to the B&R Environmental Project Manager. Samples are then logged into the laboratory's laboratory information management system (LIMS). Other pertinent issues relating to sample custody, such as interlaboratory chain-of-custody procedures, and specific procedures

for sample handling, storage, dispersement for analysis, and remnant disposal, are discussed in the laboratory SOPs included in Appendix A.

5.3 FINAL EVIDENCE FILES

The B&R Environmental central file will be the repository for all documents which constitute evidence relevant to sampling and analysis activities as described in this QAPP. B&R Environmental is the custodian of the evidence file and maintains the contents of these files for the RI, including all relevant records, reports, logs, field notebooks, pictures, subcontractor reports and data reviews in a secure, limited access location and under custody of the B&R Environmental facility manager. The control file will include at a minimum:

- field logbooks
- field data and data deliverables
- photographs
- drawings
- soil boring logs
- laboratory data deliverables
- data validation reports
- data assessment reports
- progress reports, QA reports, interim project reports, etc.
- all custody documentation (tags, forms, airbills, etc.)

Upon completion of the contract, all pertinent files will be relinquished to the custody of the United States Navy.

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 as it does to fixed 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

With the exception of the field GC, field instrument calibration is discussed in Section 9.1 of the attendant Field Sampling Plan. A summary of the requirements specific to calibration of the field GC for on-site analysis of volatile organic compounds is provided in the following paragraphs.

All compounds listed in Table 1-3 will be included in the calibration standards for field GC analysis. Standard solutions for field GC analysis will be purchased as manufacturer-certified solutions, if available. Otherwise, stock solutions will be prepared from pure standard materials. Standards for field GC analysis are further discussed in Section 5.5 of the field GC SOP (Appendix C).

A five-point initial calibration is required before any samples are analyzed. The Percent Relative Standard Deviation (%RSD) for all target compounds shown in Table 1-3 must be less than or equal to 30 percent. A mid-point continuing calibration is required at the beginning and end of every 12-hour period of sample analysis or after every 20 analytical runs, whichever is more frequent. Continuing Calibration Percent Differences (%Ds) for site-specific target compounds must not exceed 25 percent. Initial and continuing calibration procedures, acceptance criteria, and corrective action are further described in Section 5.8 of the field GC SOP included in Appendix C.

6.2 LABORATORY INSTRUMENT CALIBRATION

Calibration procedures for laboratory balances and thermometers are described in SOP LTL-1005 and SOP LTL-1006, respectively, included in Appendix A. Method- and instrument-specific calibration and tuning criteria for particular analyses are described briefly below. The frequency of calibration will be performed according to the requirements of the specific methods.

6.2.1 Volatile Organic Compound Analyses

For the analysis of volatile organic compounds in aqueous samples, the GC/MS system will be tuned and calibrated in accordance with the requirements associated with a 25 mL sample volume as specified in SOP LTL-8260 (Appendix A). For the analysis of volatile organic compounds in soil samples, the GC/MS system will be tuned and calibrated in accordance with the Contract Laboratory Program Statement of Work (OLM03.1). For either matrix, a bromofluorobenzene (BFB) instrument performance check (tuning check) must be run prior to the initial and each continuing calibration and must meet all method-specified criteria before analyses may continue. Initial calibration is required before any samples are analyzed and must include a blank and a minimum of five different concentrations as specified in the methods. A continuing calibration check, including the mid-range standard and a blank, must be performed at the beginning of each 12-hour shift during which analyses are performed.

6.2.2 Semivolatile Organic Compound Analyses

For semivolatile organic compounds, the GC/MS system will be calibrated in accordance with the CLP SOW (OLM03.1). A decafluorotriphenyl phosphine (DFTPP) instrument performance check (tuning check) must be run prior to the initial and each continuing calibration and must meet all method-specified criteria before analyses may continue. Initial calibration is required before any samples are analyzed and must include a blank plus five different concentrations as specified in the method. Standards for pyridine will be included in the initial and continuing calibrations at concentrations specified by the SOW for semivolatile compounds. A continuing calibration check, including the mid-range standard and a blank, must be performed at the beginning of each 12-hour shift during which analyses are performed.

6.2.3 PCB Analyses

For PCB analyses, the GC system will be calibrated in accordance with the CLP SOW (OLM03.1) with some modifications since only PCBs, and not pesticides, are being analyzed. Initial calibration is required before any samples are analyzed. The initial calibration and calibration verification procedures and frequencies will be performed as described in SOP LTL-8082 and the Addendum to SOP LTL-8082 (Appendix A).

6.2.4 Metals Analyses

6.2.4.1 Inductively Coupled Argon Plasma (ICP) Analyses

Inductively coupled plasma spectrometry (ICP) systems will be calibrated in accordance with the EPA CLP protocols outlined in ILM04.0. Initial calibration is required each day before any samples are analyzed and consists of a calibration blank and at least one standard. Following initial calibration, an initial calibration verification sample (obtained from a different source than the solutions used for calibration), an initial calibration blank, and interference check samples are analyzed. A continuing calibration verification sample and a continuing calibration blank are run every 2 hours or every 10 samples, whichever occurs first. Interference check samples must be analyzed a minimum of twice per 8-hour working shift. A continuing calibration verification sample, a continuing calibration blank, and interference check samples are also run after analysis of the last sample.

If any of the continuing calibration samples fail to yield a response within 10% of the true value, initial calibration will be repeated, and all field samples analyzed since the last in-control calibration standard will be reanalyzed.

6.2.4.2 Furnace Atomic Absorption Analyses

Furnace atomic absorption analyses will be calibrated in accordance with the EPA CLP protocols outlined in ILM04.0. Initial calibration is required each day before any samples are analyzed and consists of a calibration blank and at least three calibration standards covering the range of concentrations of interest. The correlation coefficient of the regression of concentration versus response should be 0.995 or greater. Immediately following initial calibration, an initial calibration verification sample (obtained from a different source than the solutions used for calibration) and an initial calibration blank are analyzed. A continuing calibration verification sample and a continuing calibration blank are run every two hours or every ten samples, whichever occurs first. A continuing calibration verification sample and a continuing calibration blank are also run after analyses of the last sample.

If any of the continuing calibration samples fail to yield a response within 10% of the true value, initial calibration will be repeated, and all field samples analyzed since the last in-control calibration standard will be reanalyzed.

6.2.5 Engineering/Biological/Miscellaneous Parameters

Calibration and standardization requirements for the remaining required parameters are described in the applicable SOPs included in Appendix A.

7.0 ANALYTICAL AND MEASUREMENT PROCEDURES

Geoprobe groundwater samples, monitoring well groundwater samples, soil samples, and field Quality Control samples (e.g., trip blanks, rinsate blanks, etc.) collected during the NIROP Fridley Operable Unit 3 (OU3) Remedial Investigation (RI), will be analyzed by Laucks Testing Laboratories, Inc., 940 South Harney Street, Seattle, Washington 98108; (206) 767-5060; FAX (206) 767-5063. The laboratory maintains Standard Operating Procedures (SOPs) for all required analyses. Analysis for dissolved methane will be performed by Microseeps, 220 William Pitt Way, Pittsburgh, PA 15238; (412) 826-5245; Fax (412) 826-3433.

The analytical methods to be used for analysis of the OU3 RI samples have been selected based on existing information regarding the NIROP plant building. During a previous investigation at the East Plating Shop, various metals, cyanide, volatile and semivolatile organics, PCBs, and petroleum hydrocarbons were detected in environmental matrices (soil and/or groundwater). Furthermore, based on the industrial nature of operations at the facility, it is possible that multiple types of chemicals could have been released via drywells, sumps, etc.

Although the presence of volatile organic constituents in the groundwater is a primary concern for the facility, information regarding the types of chemicals released is currently insufficient to develop a focused analytical program. Therefore, the suite of analyses for the OU3 RI is comprehensive and is inclusive of TCL volatiles, semivolatiles, and PCBs as well as TAL metals and cyanide. The only CLP analytical fraction not planned for analysis is the TCL pesticides fraction. Based on the nature of operations at the facility, there is no reason to believe that pesticides will be present in the soil or groundwater beneath the plant.

The NIROP OU3 RI is focused on source characterization. Based on existing analytical data for both the East Plating Shop and downgradient groundwater, it is anticipated that concentrations will be relatively high in source areas within the building. A low-level EPA method was chosen for the analysis of volatile organics in aqueous samples, since these compounds are of primary concern at the site. Standard CLP and EPA methods were chosen for the remaining parameters. With the exceptions noted in Section 7.2.1, the Contract Required Quantitation and Detection Limits (CRQLs and CRDLs) will be adequate for these parameters for the purposes of source characterization. Field measurements and analytical procedures are discussed in more detail in the remainder of this section.

7.1 FIELD MEASUREMENT PROCEDURES

Field measurements to be completed during the OU3 field investigation will include those completed in support of health and safety considerations, well development and purging, general chemical and physical characterization of groundwater, selection of soil samples for laboratory analysis, and evaluation of the natural attenuation of chlorinated solvents in groundwater. Chemical/physical parameters to be measured using field instrumentation or field test kits include volatile organics as methane equivalents, temperature, specific conductance, hydronium ion concentration (pH), dissolved oxygen, turbidity, oxidation-reduction potential, dissolved ferrous iron, dissolved reduced manganese, and hydrogen sulfide (groundwater samples). Measurement of field parameters is discussed in Section 9.0 (Field Measurements/Screening) of the Field Sampling Plan provided as Volume II of this deliverable. Calibration of field instruments is discussed in Section 9.1 of the Field Sampling Plan. Analysis for volatile organic compounds on-site using a field GC will also be performed. A SOP for field GC analysis is included in Appendix C.

7.2 LABORATORY ANALYTICAL AND MEASUREMENT PROCEDURES

Table 7-1 provides a summary of the laboratory analytical methods and associated laboratory SOPs for the OU3 RI. With the exception of TCL volatiles in aqueous samples, all samples for TCL volatile and semivolatile organics and TAL metals and cyanide will be analyzed in accordance with the CLP analytical procedures set forth in the U.S. EPA Statement of Work for organics analysis (OLM03.1) and inorganic analysis (ILM04.0), respectively. TCL volatile organic compounds in aqueous samples will be analyzed using SW-846 Method 8260A with a 25 mL sample volume as specified in SOP LTL-8260 (Appendix A) in order to achieve lower quantitation limits. Samples for TCL PCB analysis will be analyzed in accordance with OLM03.1 with the modifications provided in the Addendum to SOP LTL-8082 (Appendix A). These modifications focus the calibration and other quality control measures on PCB analysis since pesticide analysis will not be performed. Non-CLP methods will be used for quantitation of the remaining parameters. Standard Operating Procedures for these analyses are included in Appendix A.

It should be noted that the 10% frequency requirement for matrix spikes and laboratory duplicates for inorganic analyses which is specified in this QAPP exceeds the requirements stated in the CLP SOWs and laboratory SOPs. The more stringent frequency requirement, as specified in this QAPP, will override the requirements stated in the SOWs and SOPs and must be met for the OU3 RI project samples. In addition, the laboratory will note in the data package narratives the presence of peaks during volatile or semivolatile analysis which indicate the presence of petroleum compounds.

7.2.1 List of Project Target Compounds and Detection Limits

A complete list of the target compounds/analytes, Contract Required Quantitation and Detection Limits, Practical Quantitation Limits, and laboratory method and instrument detection limits is provided in Section 1.4 of this QAPP. The method detection limits shown have been experimentally determined using Laucks Testing Laboratories, Inc., SOP LTL-1011 which is included in Appendix A and is based on the method found in 40 CFR Part 136 Appendix B (FR Vol. 49, No. 209, pages 198-199). The instrument detection limits shown have been experimentally determined as specified in the CLP Statement of Work (ILM04.0). With the exceptions noted in the following paragraph, data generated through use of CLP protocols will be reported to the Contract Required Quantitation Limit (CRQL) for organics analysis and the Contract Required Detection Limit (CRDL) for inorganics analysis. All environmental data generated through use of non-CLP methods will be reported to the analyte's Practical Quantitation Limit (PQL), if applicable. An analyte's PQL is an expression of the method detection limit with consideration given to required adjustments to ensure that precision and accuracy requirements of the method are attainable. Results for ferrous iron and sulfide spot tests will be reported as qualitatively present or absent.

Contract Required Quantitation Limits for several semivolatile organic compounds and PCBs have been revised as noted in Table 1-4. These revisions have been made to reflect the laboratory's "true" reporting limits for compounds for which the standard CRQL exceeds MPCA HRL or other state criteria.

All solid sample results will be reported on a dry-weight basis. Quantitation and detection limits will also be adjusted, as necessary, based on dilutions and sample volume.

7.2.2 List of Associated Quality Control Samples

In addition to the field quality control samples (field duplicates, trip blanks, rinsate blanks, etc.) discussed in Section 3.0 of this Quality Assurance Project Plan, laboratory quality control samples including matrix spike/matrix spike duplicate samples, method blanks, preparation blanks, laboratory control samples, etc. will be analyzed. Laboratory Quality Control samples are discussed in additional detail in Sections 3.0 and 8.0 of this QAPP.

TABLE 7-1

**SUMMARY OF ORGANIC AND INORGANIC ANALYTICAL PROCEDURES
SOLID AND AQUEOUS MATRICES
NIROP FRIDLEY, MINNESOTA
PAGE 1 OF 2**

Analytical Parameter	Analytical Method	Standard Operating Procedure (1)
TCL Volatile Organics - aqueous samples	SW-846(2) 8260A	LTL-8260 (low-level option)
TCL Volatile Organics - soil samples	OLM03.1(3)	---
TCL Semivolatile Organics	OLM03.1	---
TCL Polychlorinated Biphenyls	OLM03.1, Modified	LTL-8082 plus addendum
TAL Metals and Cyanide	ILM04.0(4)	---
Total Suspended Solids	SM(5) 2540D	LTL-9202
Alkalinity (as $CaCO_3$)	EPA(6) 310.1	LTL-9005
Hardness (as $CaCO_3$)	EPA 130.2	LTL-9009
Sulfate	EPA 300.0	LTL-9110
Nitrate	EPA 300.0	LTL-9110
Nitrite	EPA 300.0	LTL-9110
Dissolved Chloride	EPA 300.0	LTL-9110
Dissolved Bromide	EPA 300.0	LTL-9110
Dissolved Phosphate	EPA 300.0	LTL-9110
Dissolved Methane	Chapelle(7)	AM18
Total Organic Carbon	Lloyd Kahn(8)	LTL-9116
Hexavalent Chromium	SW-846 3060A/7196A	LTL-7014/LTL-7401
pH	SW-846 9045C	LTL-9113
Oxidation-Reduction Potential	ASTM Method D1498-76(9) Modified for Soil Samples	LTL-9128 plus Addendum
Ferrous Iron	Spot Test(10)	LTL-7601
Sulfide	Spot Test(11)	LTL-9205

TABLE 7-1

**SUMMARY OF ORGANIC AND INORGANIC ANALYTICAL PROCEDURES
SOLID AND AQUEOUS MATRICES
NIROP FRIDLEY, MINNESOTA
PAGE 2 OF 2**

1 Laucks Testing Laboratories, Inc., SOPs for all non-CLP analyses are included in Appendix A.
2 U.S. EPA, 1986. Test Methods for Evaluating Soil Wastes, Physical/Chemical Methods. SW-846, 3rd Ed.
3 U.S. EPA (U. S. Environmental Protection Agency) CLP, 1994. Statement of Work for Organics Analysis, Multi-Media, Multi-Concentration, OLM03.1.
4 U.S. EPA CLP, 1995. Statement of Work for Inorganics Analysis, Multi-Media, Multi-Concentration, ILM04.0.
5 M. A. H. Franson (Managing Editor). Standard Methods for the Examination of Water and Wastewater. 18th Ed.
6 U.S. EPA, 1983. Methods for Chemical Analysis of Water and Wastes.
7 Francis H. Chapelle, U.S. Geological Survey. Protocol for Assessing the Natural Attenuation of Chlorinated Ethenes in Groundwater Systems. July, 1996.
8 U.S. EPA, Region II, Environmental Services Division, Monitoring Management Branch. Lloyd Kahn, Determination of Total Organic Carbon in Sediment.
9 American Society for Testing and Materials, 1981. Standard Practice for Oxidation-Reduction Potential of Water. ASTM Designation: D1498-76.
10 Fritz Feigl. Spot Tests in Inorganic Analysis. 1958.
11 Wilfred W. Scott, Sc.D. Standard Methods of Chemical Analysis. 5th Ed., Volume 1.

8.0 INTERNAL QUALITY CONTROL CHECKS

Field-related Quality Control checks were discussed in Section 3.0 of this Quality Assurance Project Plan and in Section 8.0 of the attendant Field Sampling Plan (Volume II). This section provides additional information regarding internal quality control checks for the field and the laboratory.

8.1 FIELD QUALITY CONTROL CHECKS

Quality Control (QC) procedures for pH, specific conductance, temperature, dissolved oxygen, oxidation-reduction potential, and turbidity will include calibrating the instruments as described in Section 9.1 of the Field Sampling Plan (FSP) and in the Standard Operating Procedures provided in Appendix A of the FSP. Quality Control procedures for the field GC were discussed in Section 3.0 of this QAPP. Assessment of field sampling precision and bias will be made by collection of field duplicates and rinsate blanks for laboratory analysis. Collection of the QC samples will be in accordance with the procedures provided in Section 8.0 of the FSP at the frequencies indicated in Tables 2-3 and 2-4 of the FSP. Quality Control limits for field-related Quality Control checks were provided in Section 3.0 of this QAPP.

8.2 LABORATORY QUALITY CONTROL CHECKS

The identified subcontract laboratory (Laucks Testing Laboratories, Inc.) has a Quality Control program that ensures the reliability and validity of the analyses performed at the laboratory. The laboratory maintains a Quality Assurance Plan which describes the policies, organization, objectives, quality control activities, and specific quality assurance functions employed by the laboratory. A copy of the Table of Contents for the Laucks Testing Laboratories, Inc., Quality Assurance Plan is provided in Appendix B. In addition, several SOPs regarding laboratory Quality Assurance procedures are included in Appendix A. The Table of Contents included in Appendix A provides a list of SOP titles and associated SOP numbers.

Reagent water is produced in the laboratory by a deionizing system consisting of two mixed bed deionizers, one carbon bed, and one colloid removal bed. The water is polished by an 0.2 μm filter before being delivered to bench locations by a PVC plastic plumbing system. Reagent water is checked weekly for conductivity which must be less than 1.0 μmho/cm. Reagent water is checked monthly for the following parameters using the criteria shown:

- Total Matter - less than 2 mg/L
- Soluble Silica - less than 10 µg/L
- Ammonia - less than 100 µg/L
- Non-Purgeable Organic Carbon - less than 1.0 mg/L

The pressure drops across the filters are checked and logged weekly. The filters are changed when the pressure drop exceeds 20 psi. Tell-tale lights for conductance are checked weekly, and resin beds exchanged when the light goes out. Filter and resin bed changes, the results of all checks, and any maintenance performed by outside service engineers is recorded in the reagent water logbook.

All analytical procedures are documented in writing as SOPs. Laboratory SOPs for all non-CLP analyses are provided in Appendix A of this QAPP. Internal quality control procedures for CLP analyses (volatile and semivolatile organics, PCBs, metals, and cyanide) are specified in the Statements of Work (SOWs) for organics (OLM03.1) and inorganics (ILM04.0). Modifications to OLM03.1 for PCB analyses are provided in the Addendum to Laucks SOP LTL-8082 (Appendix A). Internal quality control procedures for all non-CLP analyses (including TCL volatile analysis for aqueous samples) are specified in the method-specific SOPs provided in Appendix A. It should be noted that the 10% frequency requirement for matrix spikes and laboratory duplicates for inorganic analyses which is specified in this QAPP exceeds the requirements stated in the CLP SOWs and laboratory SOPs. The more stringent frequency requirement, as specified in this QAPP, will override the requirements stated in the SOWs and SOPs and must be met for the OU3 RI project samples.

Several internal laboratory Quality Control checks are briefly discussed in the remainder of this section.

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. A method blank generally consists of an aliquot of analyte-free water (or purified sodium sulfate for soil/sediment samples) that is subjected to the same preparation and analysis procedures as the environmental samples undergoing analysis. With the exception of recognized VOC common laboratory contaminants (e.g., methylene chloride, acetone, 2-butanone, and phthalate esters), method blanks must not contain levels of target analytes above the reported detection limits (above 2.5X the CRQL for methylene chloride and above 5X the CRQL for acetone, 2-butanone, and phthalate esters). If method blank contamination is found to exist above allowable limits, corrective actions indicated in the CLP SOWs or laboratory SOPs must be followed.

Under no circumstances are laboratory method blank contaminant values subtracted from environmental sample analysis results.

Instrument blank analysis is performed during PCB analysis to demonstrate that PCBs are not detected at greater than 0.5 times the CRQL and that the surrogate retention times are within the retention time windows. If analytes are detected at greater than half the CRQL, or the surrogate retention times are outside the retention time windows, all data collection must be stopped and corrective action must be taken. An acceptable instrument blank must be run before additional data is collected. One instrument blank every 12 hours is the minimum contract requirement.

Matrix spike analysis for organic fraction analyses will be performed in duplicate with a frequency of one per 20 environmental samples of like matrix as a measure of laboratory precision. For inorganic analyses, matrix spike and **laboratory duplicate** analysis will be performed for every 10 environmental sample analyses of like matrix. With the exception of volatile and semivolatile MSD analyses, laboratory duplicates are prepared by thoroughly mixing and splitting a sample aliquot into two portions and analyzing each portion following the same analytical procedures that are used for the environmental sample analyses. For volatile and semivolatile MSD analyses, a second sample aliquot is used for analysis in order to avoid constituent loss through the homogenization process. The field crew provides extra volumes of sample matrices designated for laboratory quality control analyses, as required. Control limits for laboratory duplicate analyses are specified in the SOWs for CLP analyses and are established statistically by the laboratory in accordance with method-specific procedures and general protocols outlined in the laboratory SOPs for non-CLP analyses. The laboratory SOPs and CLP SOWs define under what circumstances corrective actions are warranted and how they must be performed when required.

Surrogates are organic compounds (typically brominated, fluorinated, or isotopically labeled), which are similar in nature to the compounds of concern, and which 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 are evaluated against control limits specified in the CLP SOW, where applicable, or laboratory-derived statistical control limits.

Laboratory control samples (LCS) serve to monitor the overall performance of each step during the analysis, including the sample preparation. Laboratory control sample analysis will be performed for

metals and engineering parameter analyses. Aqueous LCS results must fall within the control limits specified in the CLP SOW, where applicable, or statistically established by the laboratory. Solid LCS results must fall within the control limits established by EPA-EMSL/LV, where applicable, or the supplier of the LCS standard. Aqueous and solid Laboratory Control Samples shall be analyzed utilizing the same sample preparations, analytical methods and QA/QC procedures as employed for the samples.

Internal standard performance criteria ensure that 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 Quality Control checks include mass tuning for GC/MS analysis and second column confirmation for GC/EC analysis.

9.0 DATA REDUCTION, VALIDATION, AND REPORTING

This section describes the procedures to be used for data reduction, validation, and reporting for the Operable Unit 3 (OU3) Remedial Investigation (RI) for the NIROP Fridley. All data generated during the course of the OU3 RI will be maintained in hardcopy form by B&R Environmental in the Naval Facilities Engineering Command Southern Division central files located in Pittsburgh, Pennsylvania.

In addition to the central files, all validation reports and electronic data will be maintained in the Chemistry/Toxicology/Risk Assessment Department database records files located in Pittsburgh. A Standard Operating Procedure (SOP CT-05) governs Database Management and Quality Assurance and is included in Appendix C. Upon completion of the contract, all files will be relinquished to the United States Navy.

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 Detectors (for health and safety monitoring and to support selection of soil samples for shipment to the analytical laboratory), through onsite water quality testing for general indicator parameters including hydronium ion concentration (pH), specific conductance, turbidity, and temperature, and through the use of field instruments or field test kits for measurement of additional groundwater parameters including dissolved oxygen, oxidation-reduction potential, dissolved ferrous iron, dissolved reduced manganese, and hydrogen sulfide (as sulfide). On-site analysis of soils for volatile organic analyses using a field GC will also be performed.

Field measurements of organic vapor concentrations (parts per million on a volume/volume basis relative to methane or benzene) will be recorded in the site logbook but will not be used once the field effort is completed. Hence, no further reduction of field PID data will be completed. The remaining field parameters will be recorded in the site logbook and on sample logsheets immediately after the

measurements are taken and later encoded in the OU3 RI data base for presentation in the RI Report. If an error is made in the logbook, 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. No calculations will be necessary to reduce these data for inclusion in the RI Report.

Reduction of analytical results obtained via field GC analysis will be completed in accordance with Section 5.10.8 of the field GC SOP (Appendix C). Analytical data will be recorded in the field GC injection logbook. Individual sample results will be recorded on the raw analytical data and on summary data sheets. Figure 4 of the field GC SOP provides an example page format for the field GC injection logbook.

Field data will be entered in the electronic data base manually and the entries will be verified by an independent reviewer to make sure that no "transcription" errors occurred. Field measurements will be recorded and reported in the following units:

- Hydronium ion concentration (standard pH units)
- Temperature (degrees Celsius)
- Specific conductance (millimhos)
- Turbidity (Nephelometric turbidity units)
- Dissolved oxygen (mg/L)
- Dissolved ferrous iron (mg/L)
- Dissolved reduced manganese (mg/L)
- Hydrogen sulfide (as sulfide) (mg/L)
- Oxidation-reduction potential (mV)
- Volatile Organics by field GC analysis (μg/Kg)

Standard pH units as specified above is the negative logarithm (base 10) of the hydronium ion concentration in moles/liter. Additional aspects of field data handling are provided in Sections 9.2 and 9.3.

9.1.2 Laboratory Data Reduction

The majority of the laboratory analytical data for the OU3 RI will be generated via the U.S. EPA Contract Laboratory Program analytical methods, quality assurance requirements, and reporting procedures. Therefore, data reduction for volatile organics, semivolatile organics, polychlorinated biphenyls, metals, and cyanide will be completed in accordance with applicable laboratory SOPs and with the most current

Statements of Work for Organic and Inorganic Analysis as identified in previous sections of this Quality Assurance Project Plan. In addition to the TCL and TAL results, the contracted laboratory will also generate analytical results for several general chemistry parameters. Laboratory reduction of these analytical results will be completed in accordance with the method-specific laboratory Standard Operating Procedures included in Appendix A. Laboratory data reduction is also discussed in Section 6.2.5 of the laboratory's Quality Assurance Plan.

The laboratory's procedures for review and approval of data are presented in SOP LTL-1018 (Appendix A). These procedures are also discussed in Section 6.3 of the laboratory's QA Plan.

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

- TCL volatiles in soil - µg/kg
- TCL semivolatiles in soil - µg/kg
- TCL polychlorinated biphenyls in soil - µg/kg
- TAL metals in soil - mg/kg
- Cyanide in soil - mg/kg
- Total organic carbon in soil - mg/kg
- Hexavalent chromium in soil - mg/kg
- pH in soil - standard pH units
- Oxidation-reduction potential in soil - mV
- Ferrous iron in soil - qualitative presence or absence
- Sulfide in soil - qualitative presence or absence
- TCL volatiles in groundwater - µg/L
- TCL semivolatiles in groundwater - µg/L
- TCL polychlorinated biphenyl in groundwater - µg/L
- TAL metals in groundwater - µg/L
- Cyanide in groundwater - mg/L
- Total suspended solids in groundwater - mg/L
- Total hardness in groundwater - mg/L
- Total alkalinity in groundwater - mg/L
- Sulfate in groundwater - mg/L

- Nitrate in groundwater - mg/L
- Nitrate in groundwater - mg/L
- Dissolved chloride in groundwater - mg/L
- Dissolved bromide in groundwater - mg/L
- Dissolved phosphate in groundwater - mg/L
- Dissolved methane in groundwater - mg/L

With the exception of pH, ORP, ferrous iron, and sulfide in soil samples (which will only be used to support the data validation of hexavalent chromium), all laboratory analytical results will be presented in summary tables in the RI Report. These results will be presented as received by the laboratory with the possible exception of the elimination of false positives as a result of data validation (as discussed in Section 9.2).

Descriptive statistics may also be performed for use in describing the nature and extent of contamination and for risk assessment. These statistics, as described in the following paragraphs, include the determination of average concentrations for duplicate samples and the determination of upper 95% confidence limits.

Determination of average concentrations for duplicate samples will be necessary because duplicate samples will be collected as a Quality Control measure. Arithmetic means will be determined for duplicate samples for reporting purposes in summary tables in the RI Report. The original duplicate sample results will be presented in an Appendix to the RI Report as discussed in Section 9.3. Averages for duplicates will be determined using distinct equations which are contingent upon the analytical results for the duplicate samples. The equations to be used are as follows:

<u>Positive result for both the original and duplicate sample</u>:

Average = (Original Result + Duplicate Result)/2

<u>Nondetect for both the original and duplicate sample</u>:

Average = (Original Quantitation Limit/2 + Duplicate Quantitation Limit/2)/2

Nondetect for one sample and positive result for the other (when the quantitation limit/2 for the nondetect < positive result for the other sample):

Average = (Quantitation Limit/2 + Positive Result)/2

Nondetect for one sample and positive result for the other (when the quantitation limit/2 for the nondetect ≥ positive result for the other sample):

Average = Positive Result

Note that the preceding treatment of average results includes the handling of nondetects quantitatively as values equal to one-half the quantitation limit. This is a typical procedure for the handling of nondetects.

In the event that manipulation of the analytical data for evaluation of nature and extent of contamination or for risk assessment purposes is necessary, calculations to determine representative concentrations for the exposure assessment will be performed. Such procedures will only be necessary in the event that the results for various sampling locations are pooled to generate representative concentrations for an exposure unit. Based on the anticipated distance between sampling points, it is considered unlikely that data will be pooled (i.e., each individual sampling point will be treated separately). However, in the event that pooling of data is completed, representative concentrations will be determined using the following equations:

Normally distributed data

$$UCL = X_m + t(s / \sqrt{n})$$

Where: UCL = the upper 95% confidence limit
X_m = the arithmetic mean concentration
t = the Student's t statistic
s = the sample standard deviation
n = the number of samples

Log-normally distributed data

$$UCL = \exp(X_m + 0.5s^2 + sH/\sqrt{n-1})$$

Where:
- UCL = the upper 95% confidence limit
- X_m = the arithmetic mean concentration
- s = the sample standard deviation
- H = H statistic
- n = the number of samples
- exp = the exponential function (e)

Note that distributional assumption testing will be completed prior to use of the preceding equations. Either the Shapiro-Wilk W-Test or the Komolgorov-Smirnov test will be used to test for normality or log-normality.

Field Quality Control sample results will be included in the data base for the Fridley OU3 RI. Specifically, the analytical results for trip blanks, rinsate blanks, and ambient condition blanks will be provided. The results for field Quality Control Samples will be considered during the course of data validation (in concert with laboratory method blanks) to eliminate false positive results according to the 5- and 10-times rules specified in the Region V Standard Operating Procedures for Validation of CLP Organic and Inorganic Data and the National Functional Guidelines for Organic and Inorganic Data Review. The results for laboratory Quality Control samples such as method blanks will not be presented in the RI Report data base. In addition, only the original (unspiked) sample results for Matrix Spike/Matrix Spike Duplicate samples will be provided in the data base.

Additional aspects of laboratory data handling are provided in Sections 9.2 and 9.3. Treatment of both hardcopy and electronic data deliverables are discussed.

9.2 DATA VALIDATION

Validation of field measurements and laboratory analytical data are discussed in this section. 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 via compliance with the applicable Standard Operating Procedures. In addition, the field GC analyst will evaluate all QC results on a real-time basis, taking corrective actions when necessary as described in Sections 5.8 and 5.9 of field GC SOP (Appendix C). 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

All CLP laboratory analytical data will be subjected to validation in accordance with the Region V Standard Operating Procedures for Validation of CLP Organic and Inorganic Data and the National Functional Guidelines for Organic and Inorganic Data Review. Data validation will be completed to ensure that the data are of evidentiary quality. Particular emphasis will be placed on holding time compliance, equipment calibration, spike recoveries, and blank results, although all required elements of the validation process will be considered.

Validation of analytical data will be completed by the B&R Environmental Chemistry Department located in B&R Environmental's Pittsburgh office. Final review and approval of validation deliverables will be completed by the Department's Data Validation Coordinator. The analytical results for non-CLP parameters will be validated versus the methods and SOPs included in Appendix A. Validation of these data will conform to the National Functional Guidelines to the greatest extent practicable. B&R Environmental will complete the validation process in accordance with the additional requirements outlined in Standard Operating Procedure CT-03 included in Appendix C.

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

Unless difficulties arise, all samples for field GC analysis will be analyzed within three days of collection. The field GC analyst will provide verbal results within 24 hours of analysis to the FOL for use in selecting samples for fixed-base laboratory analysis. Verbal results for soil samples will be provided on a wet-weight basis. A data summary, including solid sample results on a dry-weight basis, and narrative report will be provided by the field GC analyst to the Task Order Manager within 30 days of the last sample collection. Field logs, COC reports, QC summaries (for calibration, internal standards, and matrix spikes), and individual raw data runs will also be included with the narrative report.

Field data will be reported in the units discussed in Section 9.1.1. The RI Report will include a comprehensive data base including all field measurements (specifically pH, specific conductance, temperature, turbidity, dissolved oxygen, oxidation-reduction potential, dissolved ferrous iron, dissolved reduced manganese, and field GC volatile results). Field Measurements will be transferred from the site logbook or sample logsheets to the electronic data base manually and will be reviewed for accuracy by an independent reviewer.

All records regarding field measurements (i.e., field logbooks, sampling logbooks, and sample logsheets) will be placed in the Southern Division central files upon completion of the field effort. Entry of these results in the data base 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 of the RI Report at a minimum and may also be reported in summary fashion if they are indicative of the presence of contamination (e.g., high specific conductance readings).

The B&R Information Management Systems Department will hold responsibility for field data reporting subject to oversight by the Department Manager. Key data handling personnel within the Department include the Department Manager and the Information Management Systems Group Leader.

9.3.2 Laboratory Data Reporting

Data reported by the laboratory for all analytical fractions will be in accordance with CLP reporting format, including all non-CLP data (to the extent practicable). SOP LTL-4201 (Appendix A) specifically identifies the information that will be included in CLP-type packages for organics and general chemistry parameters.

Note that based on the modifications described in the Addendum to SOP LTL-8082 (Appendix A) for TCL PCB analysis, certain summary forms related to pesticide analysis are not applicable for PCB analysis and will not be provided. All pertinent quality control data including raw data and summary forms for blanks, standards analysis, calibration information, etc., will be provided for the non-CLP analyses. Case narratives will be provided for each Sample Delivery Group.

Environmental and field Quality Control sample results (trip blanks, duplicates, rinsate blanks, ambient condition blanks) will be included in the RI Report as an appendix. The data base will include pertinent sampling information such as sample number, sampling date, general location, depth, and survey coordinates (if applicable). Sample-specific detection limits will be reported for nondetected analytes. Units will be clearly summarized in the data base and will conform to those identified in Section 9.1.2.

The analytical data will also be reported in summary fashion within the body of the RI Report text in tabular and graphic fashion. Tabular summaries will report the frequency of detection, mean concentrations, representative concentrations (if applicable), standard deviations, etc. in accordance with the data reporting requirements outlined in Risk Assessment Guidance for Superfund - Human Health Evaluation Manual (Part A). The tabular summaries will include only those analytes that are detected in at least one sample. In the event that graphical portrayals of data are informative, isoconcentration contours or "tag maps" including the location and concentration of specific Chemicals of Potential Concern will be provided in the RI Report. Quality assurance information, including surrogate recoveries, spike recoveries, spike duplicate RPDs, duplicate RPDs, and blank results will also be included in tabular form in the RI report.

Data will be handled electronically pursuant to the electronic deliverable requirements specified in B&R Environmental's Basic Ordering Agreement with analytical laboratories. This agreement requires the analytical laboratories to provide data in both hardcopy and electronic form (DBF files). The original electronic diskettes and data validation reports are maintained in the Southern Division central files. All other pertinent information, including field logbooks, sampling notebooks, chain-of-custody forms, etc. are also maintained in the central files. Various aspects of field documentation are discussed in detail in Section 5.1 of the Field Sampling Plan (Volume II of this deliverable). Standard Operating Procedure CT-05 discusses data base management and Quality Assurance and is included in Appendix C.

Validation will be completed using the hard copy data. Upon completion of validation of a Sample Delivery Group and review by the Data Validation Coordinator, the validation qualifiers will be entered in the electronic data base and will be subjected to independent review for accuracy. During this review

process, the electronic data base printout will also be contrasted with the hard copy data (Form Is) to ensure that the hard copy data and electronic data are consistent.

The B&R Information Management Systems Department will hold responsibility for laboratory data reporting subject to oversight by the Department Manager. Key laboratory data handling personnel include the Department Manager and the Information Management Systems Group Leader (Information Management Systems Department), and the Data Validation Coordinator (Chemistry Department). It is not currently planned that copies of the data validation deliverables will be provided to either the MPCA or U.S. EPA Region V. However, a summary of the validation results (actions taken and completeness, precision, and accuracy) will be provided in the RI Report.

10.0 PERFORMANCE AND SYSTEM AUDITS

Performance and system audits will be performed periodically to ensure that work is being implemented in accordance with the approved Project Plans and in an overall satisfactory manner. Such audits will be performed by various personnel and will include evaluation of field, laboratory, data validation, and data reporting processes. Examples of pertinent audits are as follows:

- The Field Operations Leader (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.

- Performance and system audits for the laboratory will be performed regularly, by a U.S. Navy Contractor in accordance with the requirements of the Navy, and in accordance with the Laboratory Quality Assurance Plan.

- Data validators will review (on a timely basis) the chemical analytical data packages submitted by the laboratory. The data validators will check that the data were obtained through use of the approved methodology, that the appropriate level of QC effort and reporting was conducted, and whether or not the results are in conformance with QC criteria. On the basis of these factors, the data validator will generate a report describing data limitations, which will be reviewed internally by the Data Validation Coordinator prior to submittal to the Task Order Manager.

- The Task Order Manager 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. Similarly, the Task Order Manager will interface with the Risk Assessment and Modeling Coordinators, as applicable.

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 Audits

10.1.1.1 Internal Field Audit Responsibilities

An independent performance and system audit of field activities will be conducted by the B&R Environmental Quality Assurance Manager (QAM) or designee. When the formal field audit is conducted, 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. An internal audit of office procedures will also be conducted by the QAM (or designee) to ensure compliance with SOPs regarding review of deliverables, verification of calculations, data handling and transcription, and recordkeeping.

10.1.1.2 Internal Field Audit Frequency

Internal field and office audits are conducted once per annum unless the complexity of the project dictates a greater audit frequency. One audit per annum is considered appropriate for the NIROP Fridley OU3 RI/FS. Based on uncertainties regarding project plan approval, mobilization cannot be pinpointed at this time. However, the field and office audits will be completed in accordance with the following milestone schedule: (1) field audit - within one month of mobilization; (2) office audit - within three months of receipt of the final analytical data package from the subcontract laboratory.

10.1.1.3 Internal Field Audit Procedures

The field and office audits will be conducted by the QAM (or designee) 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 D.

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

- Field audits will include a review of required project documentation (logbooks, sample log sheets, etc.) for completeness and agreement; and field operations (well installation, groundwater sampling, sample handling and preservation, etc.) to determine compliance with applicable SOPs.

- File audits will consist of reviewing required project records for completeness, organization, and ease of retrieval.

- Office audits will focus on compliance with Standard Operating Procedures governing deliverable review, verification of calculations, recordkeeping procedures, and data handling, transcription, and reporting.

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

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

- The auditor will generate a formal audit report which will address corrective actions. This report will be provided by the auditor to the Task Order Manager.

- The Task Order Manager will ensure that all corrective actions are addressed and will provide written verification of corrective action implementation by the auditor.

- The auditor will manage corrective action verification and audit closure providing all documentation to the QAM.

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

  - Original monitoring schedules and revisions
  - 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 Standard Operating Procedures and project planning document requirements are followed to the letter. If problems are identified, corrective action is 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 Minnesota Pollution Control Agency (MPCA), the U.S. EPA Region V, or both. Details regarding the responsibilities of these agencies, frequency, and procedures are left to the discretion of the agencies.

10.1.2.1 External Field Audit Responsibilities

At the discretion of the MPCA and U.S. EPA Region V.

10.1.2.2 External Field Audit Frequency

At the discretion of the MPCA and U.S. EPA Region V.

10.1.2.3 Overview of External Field Audit Process

At the discretion of the MPCA and U.S. EPA Region V.

10.2 LABORATORY PERFORMANCE AND SYSTEMS AUDITS

Internal and external laboratory performance and systems audits are discussed in this section.

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's QA/QC Officer performs routine internal audits of the laboratory. Internal laboratory audits are also conducted by the U.S. Navy. B&R Environmental holds no responsibility for such audits. Performance and system audits of laboratories are coordinated through the NFESC by an independent Quality Assurance contractor. It is the responsibility of the NFESC and their contractor to ensure that the contracted 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

The subcontract laboratory conducts internal system audits of each laboratory analytical department on an annual basis, at a minimum. Internal audits are performed biannually if no external audits are conducted. In addition, each laboratory department analyzes blind performance evaluation samples as described in SOP LTL-1009 (Appendix A). Data audits are also performed by the 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 on an 18-month schedule.

10.2.1.3 Internal Laboratory Audit Procedures

The laboratory QA/QC Officer conducts internal systems audits in order to detect any problems in sample flow, analytical procedures, or documentation and to ensure adherence to the good laboratory practices as described in Laucks Testing Laboratories, Inc., SOPs. Laucks Testing Laboratories, Inc., internal audit procedures are described in SOP LTL-1017 (Appendix A) and in Section 10 of the laboratory's Quality Assurance Plan.

Performance of the laboratory's internal system audits conducted while OU3 RI samples are being analyzed will be noted in the RI report. If significant problems are noted during the laboratory's internal audits, these issues, as well as any corrective actions taken, will be described.

Internal U.S. Navy laboratory audit procedures fall under the domain of the NFESC and its contractor. Procedures will be provided to the MPCA and U.S. EPA upon request.

10.2.2 External Laboratory Audits

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

10.2.2.1 External Laboratory Audit Responsibilities

It is the responsibility of the MPCA and U.S. EPA Region V to conduct laboratory audits at their discretion.

10.2.2.2 External Laboratory Audit Frequency

An external laboratory audit may be conducted by U.S. EPA Region V or MPCA prior to the initiation of the sampling and analysis activities.

10.2.2.3 External Audit Procedures

External audit procedures are at the discretion of U.S. EPA Region V and the MPCA. External laboratory audits may include (but are not limited to) review of laboratory analytical procedures, laboratory onsite audits, and/or submission of performance evaluation samples to the laboratory for analysis.

11.0 PREVENTIVE MAINTENANCE PROCEDURES

Measuring equipment used in environmental monitoring or analysis for the NIROP Fridley OU3 RI 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 are discussed in the remainder of this section.

11.1 FIELD EQUIPMENT PREVENTIVE MAINTENANCE

Preventive maintenance of field equipment is described in Section 10.1 of the attendant Field Sampling Plan (Volume II). The B&R Environmental 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 log book including a description of the symptoms and corrective actions taken. If problem equipment is detected or should require service, the equipment should be logged, tagged, and segregated from equipment in proper working order. Use of the instrument will not be resumed until the problem is resolved.

Preventive maintenance for the field GC will be the responsibility of the field GC analyst. A schedule of preventive maintenance for the field GC is provided in Section 5.7.6 of field GC SOP (Appendix C).

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.

11.2.1 Major Instruments

Table 11-1 provides a summary of preventive maintenance procedures performed by Laucks Testing Laboratories, Inc., for key analytical instruments.

TABLE 11-1

**PREVENTIVE MAINTENANCE FOR ANALYTICAL INSTRUMENTS
NIROP FRIDLEY, MINNESOTA
PAGE 1 OF 2**

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 sample lines, replace trap, replace column, clean source, replace fittings, change sample block on autosampler, replace filaments.	As needed.
GC/MS - Semivolatiles	Change injection port liner and septum, clip 5-10 cm from front of column, ramp GC oven twice to 300 C.	Daily or as needed.
	Vacuum computer's air filters.	Approx. annually.
	Clean source.	As needed.
GC	Swab EC detectors for radioactivity.	Semi-annually.
	Change O_2 traps on gas lines.	Approx. semi-annually.
	Clean autosampler syringe.	Approx. monthly.
	Change injection port liner and septum.	Approx. every 100 injections.
	Bake system, flush injection port, clip guard column, change analytical column, change carrier hydrocarbon trap.	As needed.
ICP	Clean or change air filters.	As needed.
	Clean torch, replace nebulizer tips, replace pump tubing.	As needed.
	Check sensitivity.	Daily.
GFAA	Replace or trim capillary tubing.	As needed.
	Clean entrance windows.	As needed.
Spectro-photometer	Clean sample compartment and entrance windows.	Semiannually.
	Check wavelength calibration.	Annually.

TABLE 11-1

**PREVENTIVE MAINTENANCE FOR ANALYTICAL INSTRUMENTS
NIROP FRIDLEY, MINNESOTA
PAGE 2 OF 2**

Instrument	Preventive Maintenance	Maintenance Frequency
Ion Chromatograph	Replace pump seals.	Annually.
	Lubricate analytical pump motor.	Semiannually.
	Check chromatography module and all gas lines for leaks.	Every run.
	Clean conductivity detector cell electrodes, check cell calibration.	Monthly.
	Replace bed supports, clean columns, clean AMMS (membrane suppresser), replace autosampler pipette tip.	As needed.
TOC Analyzer	Change pump tubing.	Each run.
	Change other tubing, change furnace tubes, change LiOH tube, change tin trap, adjust optical balance, change septum, change permeation dryer tubing.	As needed.
	Change IR filter screen, change gas tubing.	Check monthly; replace as needed.

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 gas chromatographs and metals instruments are of sufficient grade to minimize fouling of the instrument. The routine use of septa, chromatographic columns, ferrules, AA furnace tubes, and other supporting supplies from reputable manufacturers will assist in averting unnecessary periods of instrument downtime. An inventory of critical spare parts is also maintained by the laboratory to minimize instrument downtime.

11.2.2 Refrigerators/Ovens

The temperatures of refrigerators used for sample storage will be monitored once daily. The acceptable range for refrigerator temperatures is 4°C ±2°C. The temperatures will be recorded on a Cold Storage Temperature Log. (See Appendix 5 of SOP LTL-1008, included in Appendix A of this QAPP.) Maintenance of the log will be the responsibility of the sample custodian. The log will contain the following information:

- Date
- Time
- Temperature
- Initials of person performing the check

Assignment of responsibilities for temperature monitoring to specific personnel does not preclude the participation of other laboratory personnel. If unusual temperature fluctuations are noted, it is the responsibility of the observer to immediately notify the person in charge of the discrepancy before the condition of the samples is compromised.

Unstable or fluctuating temperatures may be indicative of malfunctions in the cooling system. On the other hand, the instability may be due to frequent opening of the door. Regardless of the cause, such an observation must be investigated, and modifications must be made to access procedures or repairs to equipment must be made to prevent jeopardizing the integrity of the samples.

Oven temperatures are checked prior to use. The required temperature is dependent on the method to be performed. The oven temperature is recorded with the associated analytical results in a logbook designated for the analytical method.

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

Compliance with the Quality Control objectives outlined in Tables 3-1 through 3-9 of Section 3.0 will be monitored via two separate mechanisms. Precision and accuracy will be assessed through data validation in accordance with the National Functional Guidelines (to the extent practicable for non-CLP analyses). Compliance with the completeness objectives for field and laboratory data/measurement will be calculated by hand (field measurements) and electronically via a database subroutine (laboratory data). Information necessary to complete the precision and accuracy calculations will be provided in electronic and hardcopy form by the subcontract laboratory. Equations to be used for the precision, accuracy, and completeness assessment are outlined in the remainder of this section.

12.1 ACCURACY ASSESSMENT

To assure the accuracy of the analytical procedures, a minimum of 1 of every 20 samples for organic analysis and 1 of every 10 samples for inorganic analysis will be spiked with a known amount of the analyte or analytes to be evaluated. The spiked sample is then analyzed. The increase in concentration of the analyte observed in the spiked sample, because of the addition of a known quantity of the analyte, compared to the reported value of the same analyte in the unspiked sample determines the percent recovery. Daily control charts are plotted for each commonly analyzed compound and kept on matrix-specific and analyte-specific bases. The percent recovery for a spiked sample is calculated according to the following formula:

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

12.2 PRECISION ASSESSMENT

Duplicate samples (for inorganic analyses) will be prepared and analyzed at a minimum frequency of 1 per every 10 environmental samples. Duplicate samples are prepared by dividing an environmental sample into equal aliquots. Matrix spike duplicate samples (for organic analyses) will be prepared and analyzed at a minimum frequency of 1 per every 20 environmental samples. Matrix spike duplicate samples are prepared by dividing an environmental sample into equal aliquots and then spiking each of the aliquots with a known amount of analyte. The duplicate samples are then included in the analytical sample set. The splitting of the sample allows the analyst to determine the precision of the preparation and analytical

techniques associated with the duplicate samples. The relative percent difference (RPD) between the sample (or spike) and duplicate (or duplicate spike) is calculated and plotted. The RPD is calculated according to the following formula:

$$RPD = \frac{\text{Amount in Sample} - \text{Amount in Duplicate}}{0.5\ (\text{Amount in Sample} + \text{Amount in Duplicate})} \times 100$$

12.3 COMPLETENESS ASSESSMENT

Completeness is the ratio of the number of valid sample results to the total number of samples analyzed with a specific matrix and/or analysis. Following the completion of the analytical testing, the percent completeness will be calculated by the following equation:

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

The results of the data validation process and the completeness assessment will be summarized in Section 4.0 of the RI Report (Nature and Extent of Contamination). Field and laboratory completeness objectives for this project are 90 percent and 95 percent, respectively.

13.0 CORRECTIVE ACTION

Under the B&R Environmental QA/QC program, it is required that any and all personnel noting conditions adverse to quality report these conditions immediately to the Task Order Manager and Quality Assurance Manager (QAM). These parties, in turn, are charged with performing root-cause analyses and implementing appropriate corrective action in a timely manner. It is ultimately the responsibility of the QAM to document all findings and corrective actions taken and to monitor the effectiveness of the corrective measures 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 quality of 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. If warranted by the severity of the problem (e.g., if a change in the approved Project Plan documents or SOPs is required), the Navy will be notified in writing via a Field Task Modification Request (FTMR), and Navy (in conjunction with U.S. EPA Region V and MPCA) approvals will be obtained. The Field Operations Leader (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 onsite project planning documents and will be placed in the final evidence file.

Minor modifications to field activities such as a slight offset of a boring location will be initiated at the discretion of the FOL, subject to onsite approval by NIROP personnel and the onsite MPCA representative. Major modifications (e.g., elimination of a sampling point) must be obtained via an FTMR.

Corrective actions for out-of-control situations during field GC analysis are documented in the field GC logbook and in the final field GC report. The field GC SOP (Appendix C) defines out-of-control situations for field GC analysis and the appropriate corrective action procedures for these situations.

FIGURE 13-1

BROWN & ROOT ENVIRONMENTAL
FIELD TASK MODIFICATION REQUEST FORM

Client Identification Project Number FTMR 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_____

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:

- 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 (included as Appendices 1 and 2 of SOP LTL-1008, which is in Appendix A of this QAPP).

Further detail describing the system used by Laucks Testing Laboratories, Inc., to identify, document, and resolve out-of-control events is provided in SOP LTL-1008.

13.3 CORRECTIVE ACTION DURING DATA VALIDATION AND DATA ASSESSMENT

As a means of oversight, the QAM will audit a percentage of the data validation, assessment, and evaluation deliverables generated/performed. Oversight audits may also be conducted directly by the U.S. Navy personnel, or by an independent data validation firm under contract to the U.S. Navy.

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, instituting a change in work procedures, or providing additional/refresher training are possible corrective actions relevant to data evaluation activities. The Task Order Manager will be responsible for approving the implementation of corrective action.

13.4 CORRECTIVE ACTION FOR ADMINISTRATIVE ACTIVITIES

Findings identified by the conduct of office procedures and file audits may also necessitate the performance of corrective actions. Corrective actions involving file management and office procedures usually consist of correction of an isolated nonconformance or the performance of activities necessary to conform with clarified guidance.

14.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

Quality Assurance reports to management will be provided in five primary formats during the course of the NIROP Fridley OU3 Remedial Investigation. Data validation letters will be prepared on a Sample Delivery Group-specific basis and will summarize Quality Assurance issues for the subcontract laboratory data. Internal audit reports regarding compliance with Standard Operating Procedures (specifically those regarding recordkeeping and review of deliverables) and compliance with the Field Sampling Plan and Health and Safety Plan are also prepared. In addition, written weekly reports summarizing accomplishments and Quality Control/Quality Assurance issues during the field investigation will be provided by the Field Operations Leader. Finally, monthly progress reports will be provided to the Navy.

14.1 CONTENTS OF PROJECT QUALITY ASSURANCE REPORTS

The contents of the specific Quality Assurance reports are as follows. 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, etc.) the Data Validation Coordinator notifies the Task Order Manager, the Technical Program Manager, and the 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 compensational adjustments. Corrective actions are initiated at the program level.

Internal field and office audits are conducted on an annual basis for each active project. The Quality Assurance Manager (or designee) conducts the audits to ensure that projects are completed in accordance with applicable Standard Operating Procedures and project planning documents. The primary emphasis of internal office audits is to ensure that all calculations are checked, that recordkeeping is conducted in accordance with Standard Operating Procedure, and that all deliverables are subjected to peer review by experienced senior staff members. Field audits are conducted to ensure that sampling, sample shipment, recordkeeping, etc. are completed in accordance with the Field Sampling Plan and relevant Standard Operating Procedures. At the completion of such audits, the Task Order Manager is provided a Quality Assurance report that outlines the scope of the audit, any findings regarding nonconformance, recommendations for corrective action, and a proposed schedule for completion of corrective action and post-corrective action monitoring.

The Field Operations leader will provide the Task Order Manager with weekly reports regarding accomplishments, deviations from the Field Sampling Plan, upcoming activities, and a Quality Assurance summary during the course of the field investigation. In addition, monthly project review meetings are held for all active Navy CLEAN projects. Issues discussed at the project review meeting include all aspects of budget and schedule compliance, and Quality Assurance/Quality Control problems. The Task Order Manager provides a monthly progress report to the Navy which addresses the project budget, schedule, accomplishments, planned activities, and Quality Assurance/Quality Control issues and intended corrective action. Any changes to the QAPP and any staff changes that affect the project during the field work will be noted in the RI Report.

14.2 FREQUENCY OF QUALITY ASSURANCE REPORTS

As discussed in the preceding section, Quality Assurance reports are generated either frequently or infrequently contingent upon the type of Quality Assurance report generated. The following frequencies will apply for the NIROP Fridley OU3 RI: 1) Data validation QA Reports - Contingent upon SDG delivery data; 2) Internal Office Audit QA Reports - Once per annum; 3) Internal Field Audit Reports - once per annum; 4) Weekly field progress reports - weekly during the course of the field investigation; 5) Monthly Progress Reports - monthly.

14.3 INDIVIDUALS RECEIVING/REVIEWING QUALITY ASSURANCE REPORTS

Data validation Quality Assurance Reports are provided to the Task Order Manager for inclusion in the project files. In the event that major problems are observed for a given laboratory, the Program Manager, Deputy Program Manager, Quality Assurance Manager, Task Order Manager, and Laboratory Services Coordinator are provided with copies of the QA report. Copies of internal field and office audit QA Reports are provided to the Program Manager, Deputy Program Manager, and Task Order Manager. Weekly field progress reports are provided to the Task Order Manager. Monthly progress reports are provided to the Navy.

APPENDIX A

LABORATORY STANDARD OPERATING PROCEDURES

APPENDIX A

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SOP NUMBER	Title
• LTL-7014	Alkaline Digestion for Hexavalent Chromium by SW-846 Method 3060A, Revision 0
• LTL-7401	The Colorimetric Determination of Hexavalent Chromium by SW-846 7196A, Revision 0
• LTL-7601	Spot Test for the Presence of Ferrous Iron (Fe^{2+}) in Soil, Revision 0
• LTL-8082	Analysis of Organochlorine Pesticides/PCBs by USEPA CLP SOW, Including Revisions Through OLM03.1, Revision 3
•	Addendum to Laucks SOP LTL-8082, Revision 3
• LTL-8260	Determination of Volatile Organic Compounds by Method SW8260A
• LTL-9005	Alkalinity (Electrometric) Using EPA Method 310.1, Revision 1
• LTL-9009	Total Hardness by EPA 130.2, Revision 0
• LTL-9110	Determination of Anions by Ion Chromatography by Method 300.0A, Revision 4
• LTL-9113	pH in Soil and Waste Samples Using SW-846 Method 9045C, Revision 2
• LTL-9116	The Determination of TOC in Soil/Sediment Samples Using 9060, Revision 1
•	Addendum to Laucks SOP LTL-9116, Revision 1
• LTL-9128	Procedure for the Determination of the Oxidation-Reduction Potential in Water or Soils, Revision 0
• LTL-9202	Total Suspended Solids (Non-Filterable Residue) Using EPA Method 160.2 / Standard Methods 2540D and Total Volatile Suspended Solids Using Standard Methods 2540E, Revision 2
• LTL-9301	Spot Test for the Presence of Sulfide in Soil, Revision 0
• AM18	Analysis of C_1 - C_4 Hydrocarbons in Water
• LTL-1002	Document Tracking and Control
• LTL-1003	Chain-of-Custody and Documentation Procedures
• LTL-1004	Documentation of Analyst Competence and Training
• LTL-1005	Analytical Balances
• LTL-1006	Refrigerator, Freezer, and Oven Thermometer Calibration and Maintenance
• LTL-1007	Maintaining Instrument Records and Logbooks
• LTL-1008	QC Corrective Action
• LTL-1009	Blind Spike Program
• LTL-1011	Procedures for the Determination and Reporting of Detection Limits, Reporting Limits, Precision and Accuracy Studies, and Control Limits
• LTL-1012	Solvent QC Monitoring for Trace Residue Analysis
• LTL-1013	Preparation, Storage, Shelf Life and Traceability Documentation of Standards and Reference Materials
• LTL-1017	Internal Audit Procedures
• LTL-1018	Overview of Review and Approval Practices for Validatable Data Packages
• LTL-1019	Controlling, Maintaining, and Monitoring Laboratory Logbooks
• LTL-2001	Waste Segregation and Disposal
• LTL-4002	Electronic Sample Entry and Log-In
• LTL-4103	Reviewing a Sample Delivery Group (SDG) and Updating Projqc in the LIMS
• LTL-4201	Package Deliverables for all Reporting Levels
• LTL-7003	Inorganic Glass Cleaning Procedure

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-7014

Title: **Alkaline Digestion For Hexavalent Chromium By SW 846 Method 3060A**

Revision history:

Number	Date
0	06/03/97

Written by: Bill Lundberg, Inorganic Supervisor Date: 6/4/97

Approved by: Harry Romberg, QA Officer Date: 6-4-97

Approved by: Karen Kotz, Laboratory Director Date: 6/5/97

1. Introduction and Scope

1.1 Method Description

1.1.1 Method 3060A is an alkaline digestion procedure for extracting hexavalent chromium, Cr(VI), from soluble, adsorbed, and precipitated forms of chromium compounds in soils, sludges, sediments, and some industrial waste materials. To quantify total CR(VI) in a solid matrix, three criteria must be satisfied: (a) the extracting solution must solubilize all forms of Cr(VI), (b) the conditions of the extraction must not induce reduction of native Cr(VI) to Cr(III), and (c) the method must not cause oxidation of native Cr(III) contained in the sample to Cr(VI). Method 3060A meets these criteria for a wide spectrum of solid matrices. Under the alkaline conditions of the extraction, minimal reduction of Cr(VI) or oxidation of native Cr(III) occurs. The addition of Mg^{2+} in a phosphate buffer to the alkaline solution has been shown to suppress oxidation if observed. The accuracy of the extraction procedure is assessed using spike recovery data for soluble and insoluble forms of Cr(VI) (e.g., $K_2Cr_2O_7$ and $PbCrO_4$), coupled with measurement of ancillary soil properties, indicative of the potential for the soil to maintain a Cr(VI) spike during digestion, such as oxidation-reduction potential (ORP), pH, organic matter content, ferrous iron, and sulfides. Recovery of an insoluble Cr(VI) spike can be used to assess the first two criteria, and method-induced oxidation is minimal except in soils high in Mn and amended with soluble Cr(III) salts or freshly precipitated $Cr(OH)_3$. The sample is digested using 0.28M Na_2CO_3/0.5M NaOH solution and heating at 90-95°C for 60 minutes to dissolve the Cr(VI) and stabilize it against reduction to Cr(III). After digestion the Cr(VI) is quantitated using SW 846 7196A.

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 using devices and placed in containers that do not contain stainless steel (e.g., plastic or glass). A 16 oz glass jar will be required due to the possible need to analyze the sample for other parameters should the matrix spike (MS) exceed limits.

1.2.2 Samples should be stored field-moist at 4°C ± 2°C until analysis.

1.2.3 Hexavalent chromium has been shown (interlaboratory studies) to be quantitatively stable in field-moist soil samples for at least one month from sample collection. In addition, Cr

(VI) has also been shown (interlaboratory studies) to be stable in the alkaline digestate for up to 96 hours after extraction from soil.

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 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 run of soils for Cr(VI) analyzed on June 2, 1997 would be A060297_CR6_S02.

1.3.3 Blank spike - A background free matrix (clean sand for soils/sediments) to which a known amount of Cr(VI) is added each time samples are prepared. Blank spikes are required on all HAZWRAP and NFESC work. Note that an LCS or SRM will substitute as a blank spike for most inorganic analyses. At this time there is no known Cr(VI) LCS. 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 DIW - Deionized water - Lab reagent water. This water should be free of virtually all analytes.

1.3.5 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.

1.3.6 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.

1.3.7 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.

1.3.8 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.

2. Equipment List and Standards

2.1 Apparatus

2.1.1 Beakers: borosilicate glassware, 250-mL, with watch glass covers.

2.1.2 Graduated Cylinder: 100-mL.

2.1.3 Volumetric Flasks: Class A glassware, 1000-mL and 100-mL with stoppers.

2.1.4 Filtration Apparatus.

2.1.5 Filter membranes (0.45 µm). Preferably cellulosic or polycarbonate membranes.

2.1.6 Heating Device - capable of maintaining the digestion solution at 90 - 95°C with continuous auto stirring capability or equivalent.

2.1.7 Volumetric pipettes: Class A glassware, assorted sizes, as necessary.

2.1.8 Calibrated pH meter.

2.1.9 Calibrated balance.

2.1.10 Thermometer (NIST-Certified or equivalent) or other appropriate temperature sensing device.

2.2 Standards

2.2.1 Potassium Dichromate, $K_2Cr_2O_7$, spiking solution, 100 mg/L Cr (VI). Dissolve 0.2829 g of dried (105°C) $K_2Cr_2O_7$ in distilled deionized water in a 1 liter volumetric flask and dilute to the mark. Store at 20-25°C in a tightly sealed container for up to six months.

2.2.2 The Blank Spike, and MS are prepared by adding 1.0 mL of the 100 mg/L standard to their respective beakers.

2.2.3 Lead Chromate: $PbCrO_4$, analytical reagent grade. The insoluble matrix spike is prepared by adding 10-20 mg $PbCrO_4$ to a separate aliquot. Store under dry conditions at 20-25°C in a tightly sealed container.

2.3 Reagents

2.3.1 Nitric acid: HNO_3 concentrated, analytical reagent grade or spectrograde quality. Store at 20-25°C in the dark. Discard if the solution has a yellow tinge; this is indicative of photoreduction of NO_3^- to NO_2^-

2.3.2 Sodium carbonate: Na_2CO_3, anhydrous, analytical reagent grade. Store at 20-25°C in a tightly sealed container.

2.3.3 Sodium hydroxide: NaOH, analytical reagent grade. Store at 20-25°C in tightly sealed container.

2.3.4 Magnesium Chloride: $MgCl_2$ (anhydrous), analytical reagent grade. 392.18 mg $MgCl_2$ is equivalent to 100 mg Mg^{2+}. Store at 20-25°C in a tightly sealed container.

2.3.5 Phosphate Buffer: 0.5M K_2HP0_4/0.5M KH_2PO_4 buffer at pH 7: Dissolve 87.09 g analytical reagent grade K_2HP0_4 and 68.04 g analytical reagent grade KH_2PO_4 in 700 mL of deionized water. Transfer to a 1L volumetric flask and dilute to volume.

2.3.6 Digestion solution: Dissolve 20.0 ± 0.05 g NaOH and 30.0 ± 0.05 g Na_2CO_3 in deionized water in a one-liter volumetric flask and dilute to the mark. Store the solution in a tightly capped polyethylene bottle at 20-25°C and prepare fresh monthly. The pH of the digestion solution must be checked before using. The pH must be 11.5 or greater; if not, discard.

3. Safety precautions and Waste Disposal

3.1 Safety Precautions

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

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

3.1.3 Electrical shock - All instruments present the possibility of electrical shock. The operator should take all precautions including ensuring that all instruments are operated with fully

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

3.2 Waste Disposal

3.2.1 The waste generated by this digestion are not hazardous and may discarded down the sink, while diluting with tap water. 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 Method Blanks

4.2.1 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 or 10% of the concentration of the lowest sample, whichever is greater.

4.2.2 Corrective action

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

eliminated. In all cases where blank contamination exceeds the control limit, a narrative comment must be made which documents the corrective actions taken.

4.3 Method Blank Spikes

4.3.1 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. A blank spike OR a standard reference material (SRM) must be analyzed. 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.

4.3.1.1 Corrective action

4.3.1.2 Recovery must be within the certified acceptance range or a recovery range of 80 to 120% or the sample batch must be reanalyzed.

4.4 Pre digestion Matrix Spike

Both soluble and insoluble pre-digestion matrix spikes must be analyzed at a frequency of one per batch of ≤ 20 field samples. The soluble matrix spike should be spiked with 1.0 mL of the spiking solution prepared in 2.2.1 (equivalent to 40 mg/kg Cr(VI)) or at twice the sample concentration, whichever is greater. The insoluble matrix spike is prepared by adding 10-20 mg of $PbCrO_4$ (2.2.3) to a separate sample aliquot. It is used to evaluate the dissolution during the digestion process. Both matrix spikes are then carried through the digestion process. More frequent matrix spikes must be analyzed if the soil characteristics within the analytical batch appear to have significant variability based on visual observation.

4.4.1 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. It is not always required that a matrix spike analysis be performed with each preparation/analysis batch, however, the minimum frequency for MS analysis is 1 each per 20 samples per matrix. This will be best accomplished by running one with every batch for many analyses. This matrix spike sample is used to evaluate the matrix effect of the sample upon recovery of the analytes. The recovery of spike analytes is calculated as follows:

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

where:

SS = concentration in spiked sample
S = native concentration in unspiked sample
SA = spiked added, the amount of spiking material actually added to the sample calculated on the sample basis

4.4.2 The recovery criteria are defined by SW 846 as 75% - 125%.

4.4.3 Corrective action

4.4.3.1 If the matrix spike recoveries are not within these recovery limits, the entire batch must be redigested/reanalyzed. If upon reanalysis the matrix spike is not within the recovery limits, but the LCS is within criteria specified in 4.3.1.2, information such as pH, Fe^{+2}, ORP, S_2 and TOC should be carefully evaluated, as the Cr(VI) data may be valid for use despite the perceived "QC failure." The information discussed below is provided to interpret ancillary parameter data in conjunction with data on spike recoveries.

When pre-digestion matrix spike recoveries for Cr(VI) are less than acceptance range minimum criterion (75%), this is indicative of highly reducing samples (e.g., anoxic sediments) with no measurable native Cr(VI) in the unspiked sample (assuming the criteria in 4.3.1.2 are met). Such a result indicates that the combined and interacting influences of ORP, pH and reducing agents (e.g., organic acids, Fe^{+2} and sulfides) caused reduction of Cr(VI) spikes. Oxidation-reduction potentials below the bold diagonal line on Fig. 2 of SW 846 Method 3060A (Eh/pH Phase Diagram, located in Appendix 2 of this SOP) indicates a reducing soil for Cr(VI). The downward slope to the right indicates that the Eh value, at which Cr(VI) is expected to be reduced, decreases with increasing pH. The solubility and quantity of organic constituents will influence reduction of Cr(VI). The presence of H_2S or other strong odors indicate a reducing environment for Cr(VI). In general, acidic conditions accelerate reduction of Cr(VI) in soils, and alkaline conditions tend to stabilize Cr(VI) against reduction. If spike recoveries are not within the recovery limits, the reductive nature of the sample <u>must</u> be documented.

4.5 Post Digestion Spike

4.5.1.1 One post-digestion Cr(VI) matrix spike must be analyzed per batch. The post-digestion matrix spike concentration should be equivalent to 40 mg/kg or twice the

sample concentration observed in the unspiked aliquot of the test sample, whichever is greater. Dilute the sample aliquot to a minimum extent, if necessary, so that the absorbance reading for both the unspiked sample aliquot and spiked aliquot are within the initial calibration curve. A guideline for the post-digestion matrix spike recovery is 85-115% recovery. If not achieved, consider the corrective actions/guidance on data use specified in 4.4.3.1. These digestates may contain soluble reducing agents for Cr(VI), such as fulvic acids.

4.6 Sample Duplicate

4.6.1 Criteria

4.6.1.1 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

4.6.1.2 Duplicate samples must have a Relative Percent Difference (RPD) of ≤ 20%, if both the original and the duplicate are ≥ four times the laboratory reporting limit. A control limit of ± the laboratory reporting limit is used when either the original or the duplicate sample is < four times the laboratory reporting limit.

4.6.2 Corrective action

4.6.2.1 In general, reanalysis of the samples should occur if duplicate values fail to meet these criteria. Extenuating circumstances or special considerations should be discussed with the Quality Assurance Officer.

4.6.2.2 If a trend in out of control RPD values is observed, the methods used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable reproducibility. Generally, if recoveries are in control and no analyte of interest was detected in any of the samples, no immediate action will be taken on that sample set.

If integrity of reported sample values is in doubt, re-analysis may be called for. Corrective actions should be discussed with the Quality Assurance Officer.

5. Operation procedures

5.1 Sample Analysis

5.1.1 Analysis sequence

5.1.1.1 A typical batch will consist of:

Prep Blank
Blank Spike
Sample
Sample Duplicate
Sample Spike-Soluble
Sample Spike-Insoluble
Sample Spike Soluble post digestion
Up to 19 more samples

5.2 Analytical Operation

5.2.1 Adjust the temperature setting of each heating device used in the alkaline digestion by preparing and monitoring a temperature blank (a 250 mL beaker filled with 50 mL digestion solution (2.3.6)). Maintain a solution temperature of 90 - 95°C as measured with a NIST-calibrated thermometer or equivalent.

5.2.2 Place 2.5 ± 0.10 g of the as received sample into a clean and labeled 250 mL beaker. The sample should be mixed thoroughly before the aliquot is removed.

5.2.3 Add 50 mL of digestion solution (2.3.6) to each sample. Add ≅ 400 mg of $MgCl_2$ (2.3.4) and 0.5 mL of 1.0 M phosphate buffer (2.3.5). Cover all samples with watch glasses. The Mg^{+2} is used to suppress oxidation of certain forms of Cr(III) (such as soluble forms) that can be oxidized to Cr(VI) during the procedure.

5.2.4 Stir the samples continuously (unheated) for at least five minutes using a stirring bar.

5.2.5 Heat the samples and maintain a temperature range of 90-95°C with constant stirring for 60 minutes at temperature.

5.2.6 Gradually cool each solution to room temperature and transfer it quantitatively to the filtration apparatus with distilled deionized water rinses and filter through a 0.45 µm membrane filter. Rinse the inside of the filter flask and filter pad with deionized water and transfer the filtrate and the rinses to a clean 250-mL beaker. NOTE: The remaining solid after filtration of the matrix spike should be saved for possible use in assessing low Cr(VI) matrix spike recoveries. Store the filtered solid at 4 ± 2°C.

5.2.7 Place a magnetic stirring bar into the sample digest beaker, place the vessel on a stirrer, and, with constant stirring, slowly add concentrated nitric acid solution to the beaker dropwise. Adjust the pH of the solution to 7.5 ± 0.5 and monitor the pH with a pH meter. If the pH of the digest should drop below 7.0, discard the solution and redigest. If overshooting pH 7.5 ± 0.5 occurs repeatedly, prepare a diluted nitric acid solution and repeat digestion procedure. CAUTION: CO_2 will be evolved. This step should be performed in a fume hood.

5.2.8 Remove the stirring bar and rinse, collecting the rinsate in the beaker. Transfer quantitatively the contents of the beaker to a 100 mL volumetric flask and adjust the sample volume to 100 mL (to the mark on the volumetric flask) with deionized water. Mix well.

5.2.9 The sample digestates are now ready to be analyzed. Determine the Cr(VI) concentration in mg/kg by SW-846 Method 7196A (colorimetrically by UV-Vis spectrometry)

5.3 Compound Quantification

5.3.1 The quantitation of Cr(VI) follows LTL-7401 with the following exceptions:

The calibration curve is prepared to go from 0.05-0.50 mg/L.

A 1 cm cell is used

5.3.2 The output from the UV-Vis is calculated in mg/L in 100 mL of solution. To calculate the concentration in the soil:

$$\text{Cr(VI), mg/Kg db} = \frac{A*100*\text{dilution}}{\text{Sa wt.g}*\frac{TS}{100}}$$

where A= concentration in mg/L in the digest

5.3.3 Any sample exceeding the linear range of the calibration curve should be diluted and reanalyzed.

5.3.4 If a sample displays a reading at the instrument that is at odds with the analyst's observation of the color of the sample, an aliquot of the sample should be prepared at the same dilution as the sample and analyzed just like a sample, except that instead of the color reagent, an aliquot of acetone only should be used. This is the turbidity correction for that sample and its value should be subtracted from that of the colorized sample in order to compute the final result.

6. Reports

6.1 Data Packet Organization

6.1.1 The bench sheet generated during the digestion should list the sample ID, analyst, test, date, temperatures, weights, ID of the standard, and any other pertinent information.

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, 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.

Appendix I

QC Summary Table

Laucks Testing Laboratories
Method 3060A/7196A QA Requirements and Corrective Actions

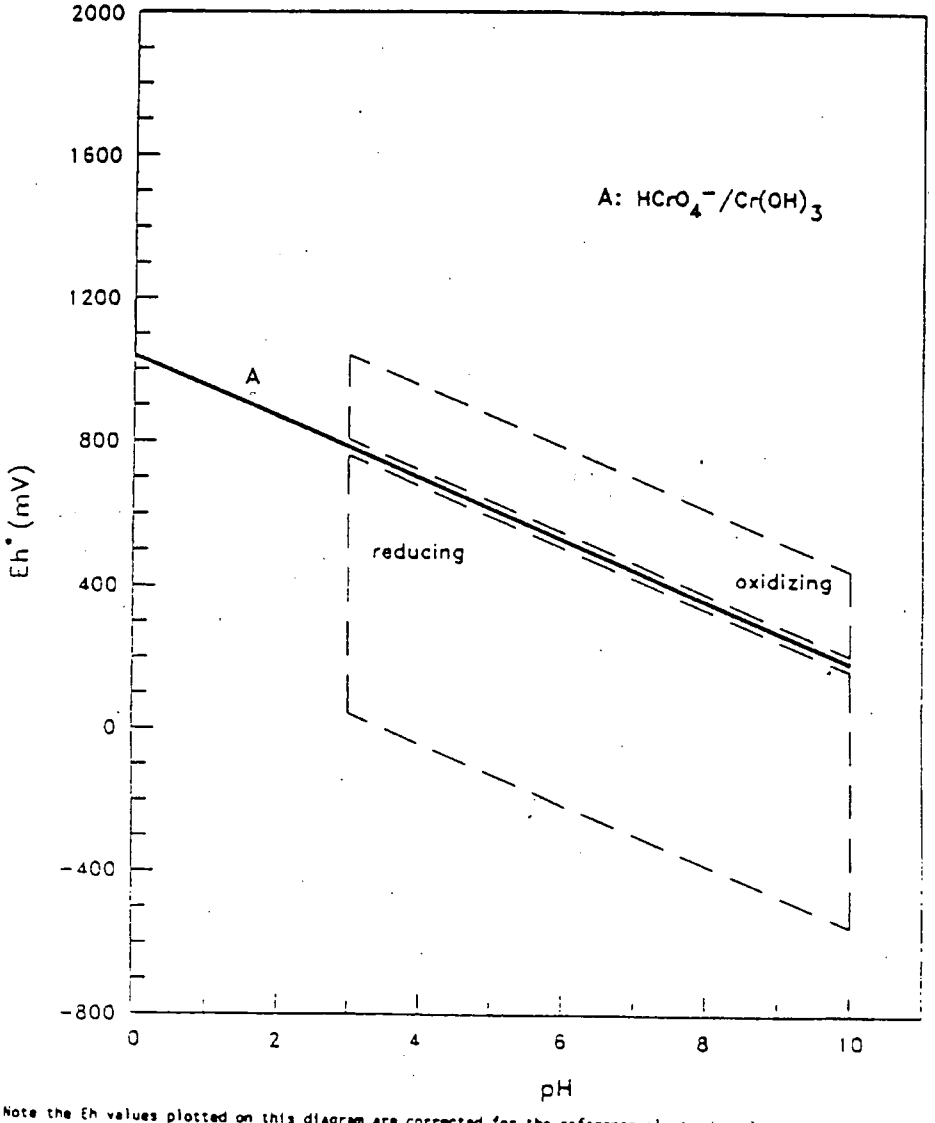
QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Method Blank	<MDL, or 10% of lowest sample	<RL, or 10% of lowest sample	1/20, minimum	Redigest	QC_DB report
Matrix Spike Recovery	75-125	75-125	1/20, minimum	Redigest	QC_DB report
Duplicate % Difference	≤20%, or ± RL	≤20%, or ± RL	1/20, minimum	Redigest	QC_DB report
Blank Spike Recovery	80-120%	80-120%	1/20, minimum	Redigest	QC_DB report
Post digestion Matrix Spike recovery	85-115%	85-115%	1/20, minimum	Redigest	QC_DB report

See text for more specifics on the corrective actions. Results may be reported if the samples are determined to cause matrix effects. However, this entails additional determinations of pH, oxidation-reduction potential and reducing agents (TOC, sulfides, ferrous iron).

Appendix II
Eh/pH Phase Diagram

FIGURE 2
Eh/pH Phase Diagram

The dashed lines define Eh-pH boundaries commonly encountered in soils and sediments.



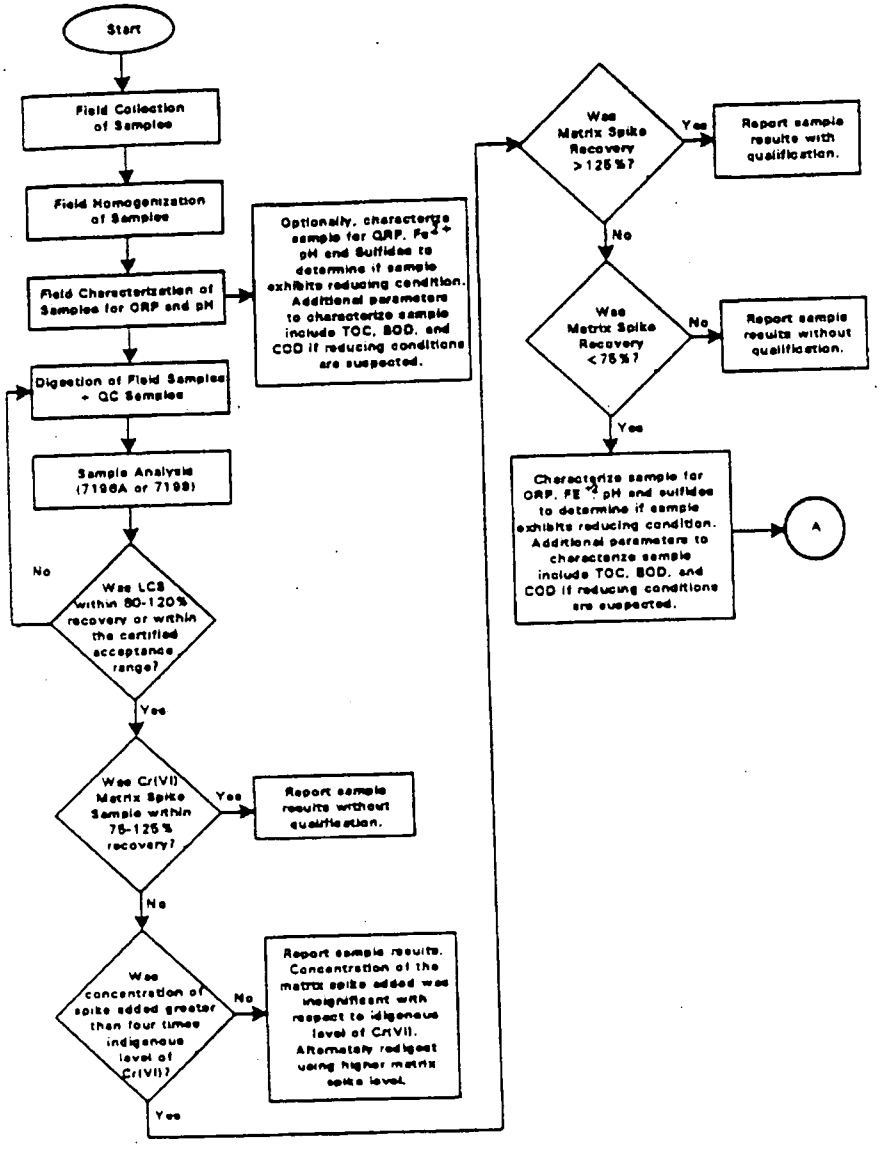
Note the Eh values plotted on this diagram are corrected for the reference electrode voltage: 244 mV units must be added to the measured value when a separate calomel electrode is used, or 199 mV units must be added if a combination platinum electrode is used.

Appendix III

Flow Charts

FIGURE 1

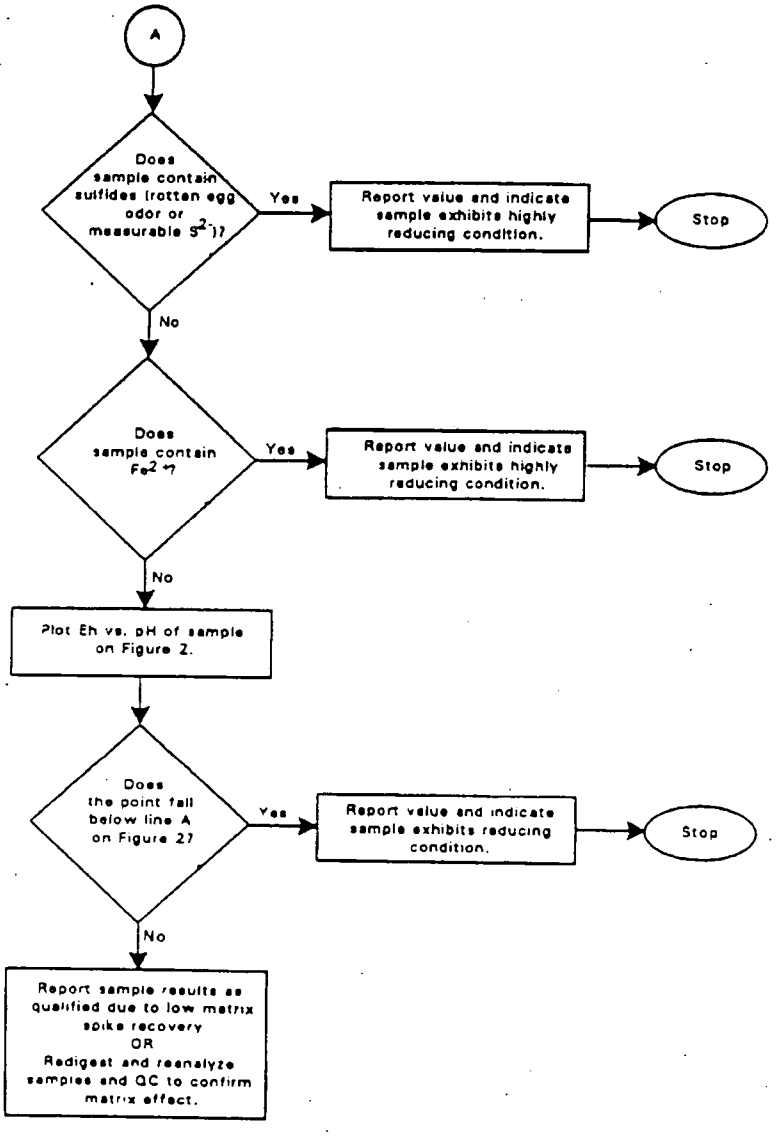
QUALITY CONTROL FLOW CHART



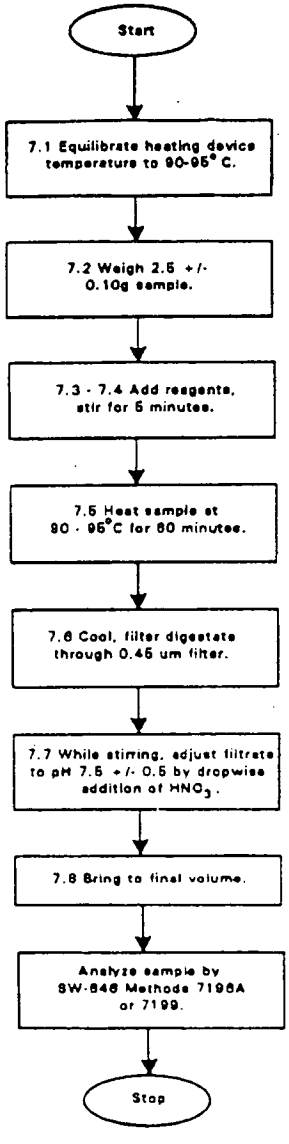
3060A-10

Revision 1
January 1995

FIGURE 1
QUALITY CONTROL FLOW CHART (Continued)



METHOD 3060
ALKALINE DIGESTION FOR HEXAVALENT CHROMIUM



LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-7401

Title: **The Colorimetric Determination of Hexavalent Chromium by SW-846 7196A**

Revision history:

Number	Date
0	02/01/96

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1. Introduction and Scope

1.1. Method Description

1.1.1. Method 7196 is used to determine the concentration of dissolved hexavalent chromium in TCLP extracts, ground waters, domestic and industrial wastes. Highly turbid or colored samples will present the possibility of positive interference, and will need to be corrected for spectrophotometrically. This method is used to measure Cr^{+6} in samples from 5-100 µg/L. Higher concentrations will require either dilution or the use of a shorter cell path.

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. Water samples should be collected in a 500 ml unpreserved bottle. Glass and plastic are acceptable. Soil samples should be collected in a 4 or 8 oz glass soil container. Samples should be stored at 4°C ± 2°C until extraction or analysis. Water samples should be analyzed within 24 hrs of collection. Soil samples should be extracted within 7 days of collection and the extract analyzed within 24 hrs.

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 second analysis for a water sample performed on Jan 2, 1996 would have the identifier A010296CR6W02.

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 (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.

1.3.6. **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. It may be considered to be a blank spike for most inorganic analyses and is preferred over artificially spiking blank materials.

1.3.16. **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. Instrument

2.1.1. This analysis uses a Perkin-Elmer Lambda 4A spectrophotometer or equivalent instrumentation. Instrumental conditions are 540 nm, slit 1, integration 2, and a 5 cm path length cell.

2.2. Standards and Reagents

2.2.1. Potassium dichromate standard solution 1, 100 µg/ml
Dissolve 0.2829 g of Primary Standard Reagent $K_2Cr_2O_7$ in DIW and dilute to 1000 ml.

This solution should be prepared annually.

2.2.2. Potassium dichromate standard solution 2, 0.10 µg/ml
Dilute 250 µl of Standard 1 to 250 mls with DIW.

This solution should be prepared fresh daily.

2.2.3. Diphenylcarbazide Solution: Dissolve 250 mg of 1,5-diphenylcarbazide in 50 mls of Reagent grade Acetone. Store this solution in an amber bottle. Discard when it becomes discolored.

2.2.4. Sulfuric Acid Solution, 10% (v/v): Dilute 10 mls of metals grade H_2SO_4 to 100 mls 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. Hexavalent chromium is highly toxic. Care should be taken to avoid ingestion.

3.2. Waste Disposal

3.2.1. All wastes from this analysis are disposed of in a laboratory sink. They should be flushed with copious amounts of water.

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 the 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. A calibration curve is prepared by measuring 0, 5, 10, 20, and 50 mls of solution 2 into 100 ml volumetric flasks. The volume is adjusted to approximately 90 mls, and then treated like the samples. The concentration of Cr^{+6} in the flasks is 0, 5, 10, 20, and 50 µg/L. The 5 µg/L standard is the reporting limit. Any samples above 50 µg/L should be diluted and reanalyzed.

The instrument is calibrated from the lowest to the highest concentration.

Due to the inherent instability of Cr^{+6}, solution 2 should be prepared fresh daily.

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 extract 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. Cr^{+6} is an inherently unstable analyte. Thus, the ICV solutions and their corresponding limits will change frequently.

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 levels of target analytes in the ICB should not exceed the reporting limit.

4.4.2. Corrective action

4.4.3. If the initial ICB 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 calibration is performed, 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.. It must be detected for the system to be considered in control.

4.5.2. Corrective Action

4.5.3. 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.

4.6. Continuing Calibration Verification (CCV) and Blank (CCB)

4.6.1. 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.

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

4.6.3. The levels of target analytes 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 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.

4.7. 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 for every 20 samples, whichever 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. Method Blank Spikes

4.8.1. 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. A blank spike or a standard reference material (SRM) should be analyzed . The SRM is the preferred material. The blank spike should only be analyzed when an SRM is not available. We currently use a material from APG.

4.8.2. Corrective action

4.8.3. 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.

4.9. Matrix Spike

4.9.1. The method requires a spike be run on every sample matrix to verify that neither a reducing condition nor a chemical interference is affecting color development. The amount of spike added should double the concentration found in the sample, and should be at least 30 ug/L 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:

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

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

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

4.9.3. Corrective action

4.9.4. 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 of a diluted aliquot of the sample. This process should also look at the recovery of at the recovery of matrix spiking compounds from the SRM and/or blank spike analysis. In all cases a narrative explanation of the condition is required to detail the corrective actions taken.

4.10. Matrix Spike Duplicate

4.10.1. 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

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

4.11. Sample Duplicate

4.11.1. Criteria

4.11.2. 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 are being performed. RPD values are calculated in a manner similar to MS/MSD RPDs:

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

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

4.11.3. 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.

4.11.4. Corrective action

4.11.5. 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 Sequence

S0
S10
S20
S50
S5
ICV
ICB/PB
Sample 1
Sample 1D (or S, depending upon client needs)
Sample 1S (or MSD, depending upon client needs)
Sample 2
Sample 3
etc
after 10 samples
CCV 1
CCB 1
Sample 11
etc
CCV 2
CCB 2

5.1. Instrumental Conditions

5.1.1. The samples are measured with a Perkin-Elmer UV-VIS spectrophotometer.

- The slit is set at 1 nm
- The integration is set for 2 secs
- The wavelength is 540 nm.
- For low level calibration use the 5 cm cells. The analytical curve may be moved up by using the 1 cm cells.
- The zero standard is used to auto-zero the instrument.

5.2. Analytical Operation

5.2.1. If Cr^{+6} in soil is requested, the sample is extracted using the TCLP extraction, SW 846 1311. This is detailed in the applicable Laucks SOP.

5.2.2. Starting with a water sample or aqueous extract:

1. Transfer 95 mls to a 100 ml volumetric flask.
2. Add 2.0 mls of the diphenylcarbazide solution and mix.
3. Adjust the pH to 2.0 ± 0.5 with 10% H_2SO_4. Use the Corning 155 pH meter, and the epopxy, gel filled electrode.
4. Dilute to 100 mls with DIW.
5. Let the solution stand for 5-10 mins for maximum color development
6. Measure the absorbance at 540 nm using the zero standard as a reference.

5.2.3. If the sample appears to be turbid, it will have to be corrected for. This is done by preparing a second aliquot. This is treated like the sample, except that the diphenylcarbazide is not added. This solution is read and the adsorbance is subtracted from the reading of the sample.

5.2.4. In some instances, the adsorbance reading will be due largely to turbidity. If the reading is greater than the high standard, the sample needs to be diluted and reanalyzed. If the client needs lower reporting limits than this can achieve:

1. Filter an aliquot of the sample, and spike an aliquot of sample and then filter. If the recovery is 85-115%, the method is in control.

2. Analyze the sample by SW 846 7197. This method is not affected by sample turbidity.

5.2.5. Compound Quantification

Cr^{+6}, mg/L = Measured concentration at instrument * dilution

If a soil sample was analyzed, the mg/L value is divided by the %Total Solids/100.

6. Reports

6.1. Data Packet Organization

6.1.1. Each data package will contain a bench sheet showing all volumes, weights, dilutions, dates and analyst's initials, a copy of the instrumental output, and a copy of the Quality Control report.

6.2. Quality Control Reports

6.2.1. All results for quality control tests are entered into the lab data base using the quality control database. A summary 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

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 II

QC Summary Table

Laucks Testing Laboratories
Method 7196A QA Requirements and Corrective Actions

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Initial Calibration	Multi-point	Multi-point R>.995	One per run	Recalibrate	Printout of calibration
Initial Calibration Verification	Required One per 15 samples	Manufacturer supplied	One per run	Recalibrate or Reanalyze	Printout of result
Initial Calibration Blank	One per batch	< Reporting limit	One per run	Reanalyze	Printout of result
Continuing Calibration Verification	Not required	90-110 % recovery	One after every 10 samples, and at the end of the run.	Recalibrate	Printout of result
Continuing Calibration Blank	Not required	< Reporting limit	One after every 10 samples, and at the end of the run.	Recalibrate	Printout of result
MDL standard recovery	Not required	Detectable	One per run	Recalibrate	Printout of result
Matrix Spike Recovery	85-115 % One per matrix	See QC database	One MS/MSD or one MS/DUP per 20	Dilute and Reanalyze	Printout of result
*MS/MSD RPD	Not Required	See QC database	One MS/MSD or one MS/DUP per 20	Dilute and Reanalyze	Printout of result
*Duplicate % Difference	Not Required	See QC database	One MS/MSD or one MS/DUP per 20	Dilute and Reanalyze	Printout of result

* Either an MSD or a Duplicate will be analyzed

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-7601

Title: **Spot Test for the Presence of Ferrous Iron (Fe^{+2}) in Soil.**

Revision history:

Number	Date
0	04/09/97

Written by: Jim Owens — Date: 4-9-97
Jim Owens, Principal

Reviewed by: Harry Romberg — Date: 4-9-97
Harry Romberg, QA Officer

Approved by: Karen J. Kotz — Date: 4/9/97
Karen Kotz, Laboratory Director

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7. References 4

1. Introduction and Scope

1.1 Method Description

1.1.1 This method covers the procedure for the qualitative determination of Ferrous (reduced) iron in soil.

2. Equipment List and Standards

2.1 Equipment

2.1.1 Test tube, 10 or 20 ml.

2.1.2 Spot plate

2.1.3 Eye dropper

2.2 Reagents

2.2.1 *a,a'*- dipyridyl solution - prepared by adding 0.1 gram of *a,a'*- dipyridyl in 10 mls of ethanol and bring to 100 ml. final volume with Type II water.

3. Safety precautions

3.1 Safety Precautions

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

4. Quality Control

4.1 Laboratory Duplicate

4.1.1 At least one sample duplicate per 10 samples is required.

4.2 Corrective Action

4.2.1 The duplicate portion of the sample should reproduce the same qualitative results as the initial aliquot. If the duplicate results do not confirm the first analysis the sample should be mixed thoroughly and two new aliquots taken for confirmation

5. Operation procedures

5.1.1 Add approximately 5 grams of representative soil to a test tube or other appropriate container. If the soil is lumpy, gently break up the sample using a mortar and pestle, if necessary.

5.1.2 Add approximately 20 mls. of deionized water and shake or mix for about one minute. Let settle until the supernatant is relatively clear, approximately 10 minutes.

5.1.3 With an eye dropper transfer approximately 1 ml. of supernatant to a spot plate. Alternately, approximately one to two grams of soil may be placed directly on the spot plate. Add several drops of the dipyridyl solution to the spot plate.

5.1.4 If ferrous iron is present a definite reddish-pink color change will develop within two to three minutes. The intensity of the color is indicative of the ferrous iron concentration.

6. Data Reporting

6.1.1 All reagent preparation must be documented in the Inorganics logbooks. All reagants must be traceable to the original stock or neat material.

6.1.2 The analyst must record the following information on the analytical benchsheet: date, analyst initials, Laucks sample identification number, sample and quality control results. Indicate the intensity of the color development, if any.

6.1.3 Copies of the above documentation must be placed in each applicable workorder file for long term document storage.

7. References

7.1.1 Spot Test in Inorganic Analysis, by Fritz Feigl, 1958.

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-8082

Title: **Analysis of Organochlorine Pesticides/PCBs by USEPA CLP SOW, Including Revisions Through OLM03.1**

Revision history:

Number	Date
1	01/28/93
2	08/15/94
3	03/10/97

UNCONTROLLED

Revised by: Cheryl DeBoer, GC Chemist Date: 3-10-97

Approved by: Karen Kotz, Laboratory Director Date: 3/10/97

Approved by: Harry Romberg, QC Officer Date: 3-10-97

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1. Introduction and Scope

1.1. Method Description

1.1.1. This document describes procedures and specifications for the instrumental analysis of chlorinated pesticides and Aroclors. The analysis is accomplished by gas chromatography utilizing a two-column electron capture detector technique. The intent of this document is to supplement the USEPA Contract Laboratory Protocol - Statement Of Work rev. OLM03.1. and as such, will mainly address optional instructions from the SOW.

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

1.2. Sample Collection, Sample Storage, Holding Times

1.2.1. Samples are normally collected in glass containers with Teflon-lined caps. All samples and sample extracts are stored at 4°C. Water samples must be extracted within 5 days of sample receipt, soil samples within 10 days of sample receipt for USEPA (for all other in-house assignments, the holding times are 7 days for water samples and 14 days for soil samples - from sample collection date). All extracts must be analyzed with 40 days of sample preparation.

1.3. Definition of Terms

1.3.1. This section defines terms and acronyms as they are used in this SOP. Other terms, such as MS/MSD or method blank, are not defined here since it is assumed that the user of this SOP already understands their more general meaning.

1.3.2. Batch Identifier — A number given to each sample delivery group which uniquely identifies that batch. This number is generally six or seven digits and is unique to the client/project.

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 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.	CRQL	Contract Required Quantitation Limit - The value used when reporting a non-detect. This is contractually set.
1.3.9.	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.
1.3.10.	%D	Percent Difference - The difference between two concentrations, expressed as a percent. Mathematically: the lower concentration is subtracted from the higher concentration, the difference is then divided by the lower concentration and that value is multiplied by 100. A %D of greater than 25% between two concentrations on different columns causes the result chosen (the lower concentration) to be flagged with a "P".
1.3.11.	PIBLK	Instrument blank. Blank solvent containing the method surrogates is injected into the instrument to monitor for carry over between sample extract injections.

1.3.12. 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.
1.3.13. RSD or %RSD	Relative standard deviation or percent relative standard deviation. The ratio of the standard deviation of a set of values to the mean of the set of values expressed as a percentage. A measure of the similarity of the values one to another.
1.3.14. RT, Retention time	The time (in minutes) at which a target analyte elutes from a chromatography column.
1.3.15. RT window	Retention time window. The +/- value which is applied to the ICV to establish the time range used to make tentative compound identifications.
1.3.16. Sequence	A set of sample extracts/digests and standard solutions introduced into an instrument in a chronologically continuous group. See also QC period.

2. Equipment List and Standards

2.1. Chromatographic System

2.1.1. A gas chromatograph with a fully programmable oven, heated injection port, autosampler, and an electronic data acquisition system capable of raw data storage.

GC system including an HP5890 GC, 7673 autosampler, 18652A or 35900A analog to digital converter, EZChrom acquisition software, and Target software, which is used for data processing.
Two each - Electron Capture Detectors (HP model 19233).
Two each - Dissimilar chromatographic columns, 0.53mm ID or 0.45mm ID, fused silica.
The lab currently has different combinations in use:

5890A: DET A; DB-608,	DET B; DB-5	0.53mmx30m
5890B: DET A; DB-5,	DET B; DB-608	0.53mmx30m
5890M: DET A; DB-5,	DET B; DB-608	0.53mmx30m

Note: Equivalent or better equipment may be substituted for the above at any time.

2.1.2. Consult maintenance logs found directly next to each instrument for details on programs and flow settings.

2.1.3. Gasses used are Helium carrier gas, Argon-5% Methane makeup gas, both high purity grades. The column carrier gas used is high purity helium with a high capacity heated water and oxygen scrubber followed with an indicator water and oxygen trap. Make-up gas is 5% Methane/Argon with high capacity water and oxygen scrubbers followed with an indicator water and oxygen trap.

2.1.4. Column flows are set at about 8.0 mls per minute. Consult individual maintenance logs for exact settings. These flows are set with an electronic bubble meter connected down stream of the detector with the make-up gas shut off at its source. Make-up gas flow is approximately 70 mls/minute.

2.1.5. All GC instruments in use are configured with an HP split/splitless injection port in the splitless injection mode. The liner is a straight through type (HP PN 19251-60540) with a small amount of silanized glass wool place just above the column end. The column is positioned 2 to 3 mm above a thick, gold plated end washer in the GC inlet.

2.2. Standards

2.2.1. Individual Standard Solution Concentrations(ng/mL) in Hexane

The standards listed below are prepared from certified materials. All working level standards are prepared in hexane (solvent), every six months, unless otherwise specified.

Pesticide Standard Mix A: Compound	INDAL STD1	INDAM STD2	INDAH STD3
1. Tetrachloro-m-xylene	5.0	20.0	80.0
2. alpha-BHC	5.0	20.0	80.0
3. gamma-BHC (Lindane)	5.0	20.0	80.0
4. Heptachlor	5.0	20.0	80.0
5. Endosulfan I	5.0	20.0	80.0
6. Dieldrin	10.0	40.0	160.0
7. Endrin	10.0	40.0	160.0
8. 4,4'-DDD	10.0	40.0	160.0
9. 4,4'-DDT	10.0	40.0	160.0
10.Methoxychlor	50.0	200.0	800.0
11.Decachlorobiphenyl	10.0	40.0	160.0
12.Isodrin	12.5	50.0	200.0

Pesticide Standard Mix B: Compound	INDBL STD1	INDBM STD2	INDBH STD3
1. Tetrachloro-m-xylene	5.0	20.0	80.0
2. beta-BHC	5.0	20.0	80.0
3. delta-BHC	5.0	20.0	80.0
4. Aldrin	5.0	20.0	80.0
5. Heptachlor epoxide	5.0	20.0	80.0
6. gamma-Chlordane	5.0	20.0	80.0
7. alpha-Chlordane	5.0	20.0	80.0
8. 4,4'-DDE	10.0	40.0	160.0
9. Endosulfan II	10.0	40.0	160.0
10.Endrin aldehyde	10.0	40.0	160.0
11.Endosulfan sulfate	10.0	40.0	160.0
12.Endrin ketone	10.0	40.0	160.0
13.Decachlorobiphenyl	10.0	40.0	160.0
14.Isodrin	12.5	50.0	200.0

2.2.2. Resolution Check Mix (ng/mL) in Hexane

Compound	Conc.
1.gamma-Chlordane	10.0
2.Endosulfan I	10.0
3.4,4'-DDE	20.0
4.Dieldrin	20.0
5.Endosulfan sulfate	20.0
6.Endrin ketone	20.0
7.Methoxychlor	100.0
8.Tetrachloro-m-xylene	20.0
9.Decachlorobiphenyl	20.0
10.Isodrin	50.0

2.2.3. Performance Evaluation Mix (PEM) (ng/mL) in Hexane
(PEM mixture is prepared weekly in Hexane.)

Compound	Conc.
1.alpha-BHC	10.0
2.beta-BHC	10.0
3.gamma-BHC(Lindane)	10.0
4.Endrin	50.0
5.4,4'-DDT	100.0
6.Methoxychlor	250.0
7.Tetrachloro-m-xylene	20.0
8.Decachlorobiphenyl	20.0
9.Isodrin	50.0

2.2.4. Pest Spike Mix solution (ug/mL) in Acetone

Compound	Conc.
1. gamma-BHC (Lindane)	0.5
2. Heptachlor	0.5
3. Aldrin	0.5
4. Dieldrin	1.0
5. Endrin	1.0
6. 4.4'-DDT	1.0

2.2.5. Surrogate Solution (ug/mL) in acetone.

Compound	Conc.
1. Tetrachloro-m-xylene	0.2
2. Decachlorobiphenyl	0.2
3. Isodrin	0.5

2.2.6. Multicomponent standard solutions (ng/mL) in Hexane

Compound	Conc.
Aroclor 1016/1260	100.0
Aroclor 1221	200.0
Aroclor 1232	100.0
Aroclor 1242	100.0
Aroclor 1248	100.0
Aroclor 1254	100.0
Toxaphene	500.0

These multi-component standards contain the following surrogates at the levels listed:

Surrogate	Conc.
Tetrachloro-m-xylene	20.0
Decachlorobiphenyl	20.0
Isodrin	50.0

2.2.7. PIBLK solution (ng/mL) in Hexane

Compound	Conc.
Tetrachloro-m-xylene	20.0
Decachlorobiphenyl	20.0
Isodrin	50.0

2.2.8. Please Refer to Appendix I for detailed listing of all stock standard mixtures.

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. The electron capture detectors contain a radioactive source and caution should be used when working with the detectors.

3.2. Waste disposal

3.2.1. The sample extracts, standards, and solvent rinses are disposed of by depositing them in the hazardous waste container located in the GC area sample preparation area - Fume hood.

3.2.2. Waste segregation and disposal from the point of collection is further covered in Laucks SOP on Watse Disposal.

4. Calibration and Quality Control

4.1. Contract Required Detection Limits

4.1.1. The CLP SOW states the following Contract Required Detection Limits for Pesticides and PCBs.

Analyte	Water (ug/L)	Soil (ug/Kg)
alpha-BHC	0.050	1.7
beta-BHC	0.050	1.7
delta-BHC	0.050	1.7
gamma-BHC (Lindane)	0.050	1.7
Heptachlor	0.050	1.7
Aldrin	0.05	1.7
Heptachlor Epoxide	0.050	1.7
Endosulfan I	0.050	1.7
Dieldrin	0.10	3.3
4,4'-DDE	0.10	3.3
Endrin	0.10	3.3
Endosulfan II	0.10	3.3
4,4'-DDD	0.10	3.3
Endosulfan sulfate	0.10	3.3
4,4'-DDT	0.10	3.3
Methoxychlor	0.50	17.
Endrin ketone	0.10	3.3
Endrin aldehyde	0.10	3.3
alpha-Chlordane	0.050	1.7
gamma-Chlordane	0.050	1.7
Toxaphene	5.0	170.
Aroclor-1016	1.0	33.
Aroclor-1221	2.0	67.
Aroclor-1232	1.0	33.
Aroclor-1242	1.0	33.
Aroclor-1248	1.0	33.
Aroclor-1254	1.0	33.
Aroclor-1260	1.0	33.

4.2. Retention Time Windows

4.2.1. Refer to the detailed discussion in Appendix V on the determination of absolute retention times as required by the CLP SOW.

4.3. Initial Multi-Point Calibration

4.3.1. Inject the standard solutions in the order specified in appendix II using evaluation criteria and corrective action specified in that appendix.

4.4. External Standard Calibration

4.4.1. External standard initial calibration data is evaluated by the %RSD.

4.4.2. CFs are calculated using the equation:

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

4.4.3. The calculated CFs are tabulated and the %RSD calculated.

4.4.1. Corrective action

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

4.5. Instrument Blank

4.5.1. After the analysis of the Initial calibration and prior to any continuing calibration verification standards, an instrument blank (PIBLK) is analyzed. This is to verify that there is no carryover between sample injections. Evaluation criteria are detailed in Appendix VII.

4.5.2. Any sample that is suspected of containing high concentrations of target analytes should be followed by a PIBLK. This PIBLK analysis is used only to make a judgment as to the possibility of carry-over into the sample extract immediately following the PIBLK.

4.6. Continuing Calibration Verification

4.6.1. The mid-range calibration standards (INDAM and INDBM) or a PEM is analyzed at the frequency detailed in the sample analysis section. In addition, these standards must be the last injection made in the analysis sequence. Evaluation criteria are detailed in Appendix IV.

4.6.2. Corrective action

4.6.2.1. The CF for each compound is calculated and the percent difference is calculated. The %D results cannot exceed the detailed CCV criteria listed in Appendix IV.

4.7. Chromatographic Resolution

4.7.1. A resolution check must be performed with every initial calibration. This check uses a separate solution, the resolution check mix (RESCHK). The resolution measured must meet the criteria detailed below.

4.7.2. Criteria

4.7.2.1. The resolution criterion is that the height of the valley between two adjacent peaks in the Resolution Check Mixture must not be greater than 60% of the height of the shorter peak. The poorest resolution on the DB-608 column probably will be between DDE and Dieldrin, between Methoxychlor and Endrin ketone and between Endosulfan I and gamma-Chlordane. On the DB-1701 column, resolution difficulties most frequently occur between Endosulfan I and gamma-Chlordane, and between Methoxychlor and Endosulfan sulfate.

4.7.3. Corrective action

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

4.8. Updating Retention Time Windows

4.8.1. The retention times for all target analytes must fall within the RT windows established by the Initial Calibration.

4.9. Instrument Blank

4.9.1. Criteria

4.9.1.1. There must be no detectable levels of target analytes in the initial PIBLK. Other PIBLKS cannot exhibit a concentration exceeding 1/2 of the CRQL.

4.9.2. Corrective action

4.9.2.1. If the initial PIBLK contains measurable levels of target analytes the system is out of control. The source of contamination must be identified and corrected. Please refer to Appendix VII for more detailed information.

4.10. Continuing Calibration Verification/Performance Evaluation Mix

4.10.1. A set of INDAM and INDBM standards or a PEM is analyzed every 12 hours.

4.10.1. Criteria

4.10.1.1. After every 12 hours a set of INDAM and INDBM standards or a PEM is analyzed. The CF for each compound is calculated and the percent difference is calculated as follows.

$$\%D = \frac{CF_i - CF_c}{CF_i} x100 \quad \text{or} \quad \%D = \frac{Ci - Cc}{Ci} x100$$

where:

CF_i = CF from ICV standard
CF_c = CF from CCV standard

4.10.1.2. The %D results cannot exceed the detailed CCV criteria.

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

4.10.2. Corrective action

4.10.2.1. 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.

4.11. Method Blanks

4.11.1. Criteria

4.11.1.1. Method blanks are used to verify contamination free reagents and apparatus. They are prepared with every set of samples extracted at the same time at least one blank every 20 samples which ever is more frequent. Any analyte response above the detection limit is reported. Method blank cannot contain any analyte at greater than the CRQL. The surrogate retention times must be within the retention time windows calculated from the initial calibration sequence, and surrogate recoveries must fall within the 30-150% recovery limits. These limits are not advisory for method blanks.

4.11.2. Corrective action

4.11.2.1. If surrogate recoveries are out of control, check all calculations. If no calculation errors are detected, reanalyze the method blank. If surrogates are still out of control, all samples associasted with the method blank must be re-extracted.

4.11.2.2. If analytes are present in the blank above the CRQL, first reanalyze the method blank. If the method blank criteria are still not met, all samples associated with the method blank must be re-extracted and reanalyzed.

4.12. Matrix Spike

4.12.1. Criteria

4.12.1.1. A sample is either chosen at random or designated by the client. An aliquot of spiking solution is added to this sample prior to extraction. It is not required that a matrix spike analysis be performed with each extraction batch, 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:

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

where:

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

4.12.1.2. Recovery control limits are detailed in Appendix VIII.

4.12.2. Corrective action

4.12.2.1. Samples with spike recoveries outside control limits will be reviewed for possible corrective action. This process should 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.

4.13. Matrix Spike Duplicate

4.13.1. Criteria

4.13.1.1. 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

4.13.1.2. RPD control limits are detailed in Appendix VIII.

4.13.2. Corrective action

4.13.2.1. Samples with spike recoveries or RPDs outside control limits will be reviewed for possible corrective action. This process should 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.

4.14. Surrogate Recovery

4.14.1. Criteria

4.14.1.1. 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

4.14.1.2. Detailed surrogate recovery control limits are tabulated in Appendix VII.

4.14.2. Corrective Action

4.14.2.1. Check calculations for possible error. Low surrogate recoveries are greater potential indicators of poor method performance than high surrogate recovery since non-GC/MS methods cannot separate co-eluting interferences. Hence corrective action is not required for high surrogate recoveries.

4.14.2.2. 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. Consult the individual maintenance log books for specific conditions. The following are general operating parameters used on gas chromatographs that are used for CLP pesticides. These conditions are maintained on 5890A, 5890B, and 5890M Gas Chromatographs.

Carrier Gas:	Helium
Column Flow:	8 mL/min
Make-up Gas:	Argon/Methane-5% (high purity)
Make-up Flow:	70 mls/min.
Injector Temperature:	205 °C
Injection:	On-column
Injection Volume:	1 µL
Injector:	Grob-type, splitless
Initial Temperature:	150 °C
Initial Hold Time:	0.5 min.
Temperature Ramp:	4 °C/min.
Final Temperature:	275 °C
Final Hold Time:	11.0 min.
ECD Temperature:	350 °C

The above conditions must be used for the analysis of all standards, samples, blanks, and MS/MSDs.

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 hoc basis based on such an observed shift. The data processing 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 Quantitation

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 CLP SOW is:

$$\text{Concentration ug/L} = \frac{(A_x)\,(V_t)\,(Df)\,(GPC)}{(CF)\,(V_o)\,(V_i)}$$

Where:

A_x = Response (area or height) of the peak for the compound to be measured.
CF = Calibration factor for the midpoint concentration external standard (area per ng).
V_o = Volume of water extracted in milliliters (mL).
V_i = Volume of extract injected in microliters (µL). (If a single injection is made onto two columns, use one half the volume in the syringe as the volume injected onto each column.)
V_t = Volume of the concentrated extract in microliters (µL).
GPC = GPC factor. GPC = 1 if no GPC clean-up was performed or GPC = 2 if GPC clean-up was performed.
Df = Dilution factor. The dilution factor for analysis of water samples by this method is defined as follows.

$$\frac{\text{µL most conc. extract used to make dilution + µL clean solvent}}{\text{µL most conc. extract used to make dilution}}$$

If no dilution is performed, Df = 1.0.

5.2.3.3. Non-aqueous samples

5.2.3.4. 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.

$$\text{Concentration (Dry weight basis)} \quad \text{ug/Kg} = \frac{(A_x)\,(V_t)\,(Df)\,(GPC)}{(CF)\,(V_i)\,(W_s)\,(D)}$$

Where:

A_x *and* CF are as given for aqueous samples above.

V_t = Volume of the concentrated extract in microliters (µL). (This volume must be 5000 µL.)

V_i = Volume of extract injected in microliters (µL). (If a single injection is made onto two columns, use one half the volume in the syringe as the volume injected onto each column.)

D = [100 x (% Moisture)] / 100

W_s = Weight of sample extracted in grams (g).

GPC = A factor used to account for the amount of extract that is lost as a result of GPC clean-up. If GPC clean-up is performed, the factor = 2. If GPC was not performed, the factor = 1. Note that GPC clean-up is <u>required</u> for all soil sample extracts.

Df = Dilution factor. The dilution factor for analysis of water samples by this method is defined as follows.

$$\frac{\text{µL most conc. extract used to make dilution + µL clean solvent}}{\text{µL most conc. extract used to make dilution}}$$

If no dilution is performed, Df = 1.0.

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. Printouts of all data entered must be included in the data packet. The routine minimum is a method blank report, a method blank spike report, and an MS/MSD report.

6.3. Sample Result Reports

6.3.1. Data Qualifying Flags

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

Code	Definition
U	The analyte of interest was not detected, to the reporting limit indicated.
B	The analyte of interest was detected in the method blank associated with the sample, as well as in the sample itself. The B flag is applied without regard to the relative concentrations detected in the blank and sample.
J	The analyte of interest was detected below the Contract Required Quantitation Limit (CRQL) but above 1/2 of the CRQL. This value should be regarded as an estimate.
D	The value reported is derived from the analysis of a diluted sample or sample extract.
P	When a dual column /dual detector GC technique is employed, this flag indicates that calculated results from the two determinations differ by more than 25%. Generally, we report the lower value.
E	The value reported is based on a sample or sample extract in which the target analyte concentration exceeded the calibration range. The value reported should be considered an estimate.
C	The target analyte's presence was confirmed by GC/MS.

6.4. Control Chart(s)

6.4.1. The recovery values for gamma-BHC (Lindane), Heptachlor, Aldrin, Aroclor 1260, Tetrachloro-m-xylene, Decachlorobiphenyl, and Isodrin in the LCS 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

7.1. USEPA CLP Statement Of Work, Revision OLM03.1, August 1994

7.2. Method for Instrumental Analysis of Organochlorine Pesticides and PCBs, Laucks Testing Labs SOP, September 1989

APPENDIX I

Standard Solution Concentrations, units

-Pesticide Matrix Spike Mix, Supelco Cat.#4-8449
-Pesticide Standard Mix A, Restek Cat.#32003
-Pesticide Standard Mix B, Restek Cat.#32004

1.1 High level A and B mixtures are made at 16 times the prescribed level of the low level standard. These standards are made in hexane every 6 months, or more frequently if the condition of the standard warrants it. Isodrin is present as an optional third surrogate at 50 ppb in the midpoint concentration levels.

1.2 Aroclors are dilutions of EPA stocks.

1.3 PEM - This standard is made from a dilution of a certified Restek stock standard; Cat.#32002.

1.4 All performance evaluation mixtures stock dilutions are made weekly in hexane.. Isodrin is present as an optional third surrogate at 50 ppb. This standard is made fresh weekly.

1.5 Resolution Check Mixture - This standard is made from a dilution of a certified Restek stock standard; Cat.#32001.

1.6 All resolution check mixture stock dilutions are made in hexane. Isodrin is present as an optional third surrogate at 50 ppb. This standard is made fresh every six months.

1.7 Surrogates are made from neat materials: DCB source is Chem Service. TCMX source is Aldrich. Isodrin source is Aldrich.

1.8 These standards are made in hexane. Isodrin is present as an optional third surrogate at 50 ppb in the midpoint concentration levels.

1.9 The supplier names and catalog numbers listed are for reference only. Certified standards from different manufacturers may be substituted at any time.

APPENDIX II

Analysis Sequence

This section is referenced to section III D (6.1 to 6.2) of USEPA CLP SOW OLM03.1 contract.

6. Initial Calibration

6.1 Initial Calibration Sequence

6.1.1 Before any samples are analyzed, it is necessary to complete the initial calibration sequence given below.

NOTE: Steps 16 and 17 are used as part of the calibration verification as well (see appendix IV).

INITIAL CALIBRATION SEQUENCE

1. Resolution Check
2. Performance Evaluation Mixture
3. Aroclor 1016/1260
4. Aroclor 1221
5. Aroclor 1232
6. Aroclor 1242
7. Aroclor 1248
8. Aroclor 1254
9. Toxaphene
10. Low Point Standard A
11. Low Point Standard B
12. Midpoint Standard A
13. Midpoint Standard B
14. High Point Standard A
15. High Point Standard B
16. Instrument Blank
17. Performance Evaluation Mixture

6.1.2 Samples may be analyzed only after the initial calibration acceptance criteria (6.2) are met. Otherwise, the analytical system is not functioning adequately for use with this protocol.

6.1.3 The initial calibration may continue to be used as long as the analytical system remains under control. The proof that the analytical system is under control is provided by the analyses of the Performance Evaluation Mixtures. If those analyses do not meet the criteria described in

appendix IV, appropriate corrective action must be taken, and the initial calibration sequence must be repeated. The calibration sequence must also be repeated if any major change in instrument hardware or instrument parameters is made (e.g., if a new column is installed or if the detector temperature is changed).

6.2 Initial Calibration Acceptance Criteria

6.2.1 The initial calibration sequence must be analyzed in the order listed in paragraph 6.1 using the GC/ECD operating conditions described in paragraph 5.1.1. The standards must be prepared according to Section 2.2 of this SOP. Calculate the calibration factors and retention times according to paragraph 9.2.2 of Appendix VI.

6.2.2 The resolution criterion is that the height of the valley between two adjacent peaks in the Resolution Check Mixture must not be greater than 60% of the height of the shorter peak. The poorest resolution on the DB-608 column probably will be between DDE and Dieldrin, between Methoxychlor and Endrin ketone and between Endosulfan I and gamma-Chlordane. On the DB-1701 column, resolution difficulties most frequently occur between Endosulfan I and gamma-Chlordane, and between Methoxychlor and Endosulfan sulfate.

6.2.3 The breakdown of DDT and Endrin in both of the Performance Evaluation Mixtures must be less than 20.0 percent, and the combined breakdown of DDT and Endrin must be less than 30.0 percent where

EQ.1

$$\% \text{ Breakdown DDT} = \frac{\text{Amount found in ng (DDD+DDE)} * 100}{\text{Amount in ng of DDT injected}}$$

EQ.2

$$\% \text{ Breakdown Endrin} = \frac{\text{Amount found in ng (Endrin Aldehyde + Endrin ketone)} * 100}{\text{Amount of Endrin injected in ng}}$$

EQ.3

$$\text{Combined \% Breakdown} = \text{\% Breakdown DDT} + \text{\% Breakdown Endrin}$$

6.2.4 All single component pesticide and surrogate peaks in both runs of the Performance Evaluation Mixtures must be greater than or equal to 90.0 percent resolved on each column.

6.2.5 The relative percent difference of the calculated amount and the true amount from each of the single component pesticides and surrogates in both of the PEMs must be less than or equivalent to 25.0 percent, using equation 4 of Appendix IV paragraph 7.1.

6.2.6 At least one chromatogram from each of the two Individual Standard Mixtures A and B, run during the initial calibration, must yield peaks that give recorder deflections of 50 to 100 percent of full scale.

6.2.7 The resolution between any two adjacent peaks in the midpoint concentrations of Individual Standard Mixtures A and B in the initial calibration must be greater than or equal to 90.0 percent.

6.2.8 The % RSD of the calibration factors for each single component analyte and surrogate must be less than or equal to 20.0 percent, except alpha-BHC and delta-BHC. The %RSD of the calibration factors for alpha-BHC and delta-BHC must be less than or equal to 25.0 percent. The %RSD of the calibration factors for the two surrogates must be less than or equal to 30.0 percent. Up to two single component target compounds (but not surrogates) per column may exceed the 20.0 percent limit for % RSD (25.0 % for alpha-BHC and delta-BHC), but those compounds must have a % RSD of less than or equal to 30.0 percent.

6.2.9 The absolute retention times of each of the single component pesticides and surrogates in both runs of the PEM must be within the retention time windows determined from the three-point initial calibration.

6.3 Corrective Action.

6.3.1 If the technical acceptance criteria for the initial calibration are not met, inspect the system for problems. It may be necessary to change the column, bake out the detector, clean the injection port, or take other corrective actions to achieve the acceptance criteria.

6.3.2 Contamination should be suspected as a cause if the detector cannot achieve acceptable linearity using this method. In the case of light contamination, baking out the detector at an elevated temperature (350 °C) should be sufficient to achieve acceptable performance. In the case of heavy contamination, passing hydrogen through the detector 1-2 hours at an elevated temperature may correct the problem. In the case of severe contamination, the detector may require servicing by the ECD manufacturer. DO NOT OPEN THE DETECTOR. THE ECD CONTAINS RADIOCHEMICAL SOURCES.

6.3.3 If the laboratory cleans out a detector using an elevated temperature, the ECD electronics must be turned off during the bake out procedure.

6.3.4 After bake out or hydrogen reduction, the detector must be recalibrated using the initial calibration sequence.

6.3.5 Initial calibration technical acceptance criteria MUST be met before any samples or required blanks are analyzed. Any samples or required blanks analyzed after the initial calibration criteria have not been met will require reanalysis.

APPENDIX III

Data Packet Check List

Organics Complete SDG FILE (CSF) Inventory Sheet (FORM DC-2-2) for PEST/PCB Data:

6. Pesticides

REQUIREMENT	DESCRIPTION	(notes, form no.)	*Check*
a.) QC SUMMARY			
Form II PEST	surrogate % recovery	water (2E)	_____
		soil (2F)	_____
Form III PEST	MS/MSD	water (3E)	_____
		soil (3F)	_____
Form IV PEST	method blank summary	(4C)	_____
b.) SAMPLE DATA			

In order by sample number & chromatograph column.
For each sample:

Form I PEST	OAD	(ID)	_____
Chromatograms:	(if no hit @ low std scale)		
first column:			
second column:			
Integration reports:	(annotated <CRQL, RT out, manual integration, etc.)		
first column,			_____
second column			_____
Manual worksheets	confirmation, etc.		_____

c.) STANDARDS DATA

Form VI	PEST-1	init. calib. single component	(6D)	_____
	PEST-2	init. calib. single component	(6E)	_____
	PEST-3	init. calib. multi component	(6F)	_____
	PEST-4	analyte resolution (form only)	(6G)	_____
	PEST-5	performance evaluation mixture	(6H)	_____
	PEST-6	individual std mixture A	(6I)	_____
	PEST-7	individual std mixture B	(6J)	_____
Form VII	PEST-I	CCVs using PEMS, PIBLKs	(7D)	_____
	PEST-2	CCVs using INDA, INDB	(7E)	_____
Form VIII	PEST	analytical sequence	(8D)	_____
Form IX	PEST-1	florisil (form only)	(9A)	_____
	PEST-2	GPC (form only, data follows)	(9B)	_____
Form X	PEST-1	ID single component	(10A)	_____
	PEST-2	ID multi-component	(10B)	_____

Chromatograms (first column, second column)
over Reports (first column, second column)
for all standards:

Resolution check data _____
PEM, _____
INDA (L,M,H), _____
INDB (L,M,H), _____
Multicomponent analytes _____
(Toxaphene, PCBS),
Midpoint INDA & INDB used as CCVs _____
Florisil data _____
GPC calibration data _____
All multicomponent standards analyzed for confirmation _____
(high level PCBS, etc.)
Integration methods for each sequence _____

d.) RAW QC DATA
In chronological order:

BLANKS:

method			
Form I PEST	OAD	(1D)	_____
Chromatograms			_____
Integration reports			_____
instrument			
Form I PEST	OAD	(ID)	_____
Chromatograms			_____
Integration reports	(annotated <1/2 CRQL)		_____
Sulfur cleanup	(soils)		
Form I PEST	OAD	(ID)	_____
Chromatograms			_____
Integration reports			_____

MATRIX SPIKE

Form I PEST	OAD	(ID)	_____
Chromatograms			_____
Integration reports			_____

MATRIX SPIKE DUPLICATE

Form I PEST	OAD	(ID)	_____
Chromatograms			_____
Integration reports			_____

MISCELLANEOUS

Analysis sequence: both channels	_____
Data reduction methods	_____
Extraction bench sheets	_____
% total solids, pH, bench sheets	_____
Logbook sheets for surrogates, spikes	_____
standards log package	_____
GPC chromatogram traces	_____

APPENDIX IV

Continuing Calibration Verification Criteria

This Appendix references section III D 9.3 of USEPA of CLP OLM03.1 SOW.

7. Calibration Verification

7.1 Three types of analyses are used to verify the calibration and evaluate instrument performance. The analyses of instrument blanks, Performance Evaluation Mixtures (PEM), and the mid point concentration of Individual Standard Mixtures A and B constitute the continuing calibration. Sample data are not acceptable unless bracketed by acceptable analyses of instrument blanks, PEMs, and both Individual Standard Mixtures A and B.

7.2 An instrument blank and the PEM must bracket one end of a 12-hour period during which sample data are collected, and a second instrument blank and the mid point concentration of Individual Standard Mixtures A and B must bracket the other and of the 12-hour period.

7.3 For the 12-hour period immediately following the initial calibration sequence, the instrument blank and the PEM that are the last two steps in the initial calibration sequence bracket the front end of that 12-hour period. The injection of the instrument blank starts the beginning of that 12-hour period. Samples may be injected for 12 hours from the injection of the instrument blank. The three injections immediately after that 12-hour period must be an instrument blank, Individual Standard Mixture A, and Individual Standard Mixture B. The instrument blank must be analyzed first, before either standard. The Individual Standard Mixtures may be analyzed in either order (A,B or B,A).

7.4 The analyses of the instrument blank and Individual Standard Mixtures A and B immediately following on 12-hour period may be used to begin the subsequent 12-hour period, provided that they meet the acceptance criteria in paragraphs 7.8-7.14. In that instance, the subsequent 12-hour period must be bracketed by the acceptable analyses of an instrument blank and a PEM, in that order. Those two analyses may in turn be used to bracket the front end of yet another 12-hour period. This progression may continue every 12 hours until such time as any of the instrument blanks, PEMS, or Individual Standard Mixtures fails to meet the acceptance criteria in paragraphs 7.8-7.14. The 12-hour time period begins with the injection of the instrument blank. Standards (PEM or Individual Standard Mixtures), samples and required blanks may be injected for 12:00 hours from the time of injection of the instrument blank.

7.5 If more than 12 hours have elapsed since the injection of the instrument blank that bracketed as previous 12-hour period, an acceptable instrument blank, and PEM must be analyzed in

order to start a new sequence. This requirement applies even if no analyses were performed since the last standard was injected.

7.6 After a break in sample analyses, the laboratory may only resume the analysis of samples using the current initial calibration for quantitation by analyzing an acceptable instrument blank and a PEM.

7.7 If the entire 12-hour period is not required for the analyses of all samples to be reported and all data collection is to be stopped, the incomplete sequence must be ended with either the instrument blank/PEM combination or the instrument blank/Individual Standard Mixtures A and B combination, whichever was due to be performed at the end of the 12-hour period.

7.8 All single component pesticides and surrogates in the Performance Evaluation Mixtures used to demonstrate continuing calibration must be greater than or equal to 90.0 percent resolved. The resolution between any two adjacent peaks in the midpoint concentrations of Individual Standard Mixtures A and B in the initial calibration must be greater than or equal to 90.0 percent.

7.9 The absolute retention time for each of the single component pesticides and surrogates in the PEMs and mid point concentration of the Individual Standard Mixtures used to demonstrate continuing calibration must be within the retention time window determined from the three-point initial calibration described.

7.10 The percent difference between the calculated amount and the true amount for each of the single component pesticides and surrogates in the PEM and mid point concentration of the Individual Standard Mixtures used to demonstrate continuing calibration must be less than or equal to 25.0 percent and greater than -25.0 percent, using Equation 4.

EQ. 4

$$RPD = \frac{|C_{nom} - C_{calc}|}{(C_{nom} + C_{calc})/2} \times 100$$

C_{nom} nominal concentration of each analyte

C_{calc} calculated concentration of each analyte from the analyses of the standard

7.11 The percent breakdown of DDT and Endrin in the PEM must be less than or equal to 20.0 percent on each column. The combined breakdown of DDT and Endrin must be less than or equal to 30.0 percent on each column.

7.12 All instrument blanks must demonstrate that no analyte may be detected at greater than ½ the CRQL for that analyte.

7.13 Analysts are cautioned that running an instrument blank and a performance evaluation mixture once every 12 hours is the minimum contract requirement. Late eluting peaks may carry over from one injection to the next if highly complex samples are analyzed or if the GC conditions are unstable. Such carryover is unacceptable. Therefore, it may be necessary to run instrument blanks and performance evaluation mixtures more often to avoid discarding data.

7.14 The requirements for running the instrument blanks, Performance Evaluation Mixture, and Individual Standard Mixtures A and B are waived when no samples, method blanks, or matrix spikes are run during that 12-hour period. After a break in sample analysis, a laboratory may resume the analysis of samples, method blanks, and matrix spikes and may use the current initial calibration for quantitation only after an acceptable PEM is run (paragraphs 7.2 - 7.6). If a successful PEM cannot be run after an interruption, an acceptable initial calibration must be run before sample data may be collected. All acceptable sample analyses must be bracketed by acceptable performance evaluation mixtures and instrument blanks.

APPENDIX V

Determination of Absolute Retention Times

This Appendix references Section III D 9.2.4 of USEPA CLP OLM03.1 SOW.

8.1 During the initial calibration sequence, absolute retention times (RT) are determined for all single response pesticides, the surrogates, and at least three major peaks of each multi-component analyte.

8.2 For single component pesticides, an RT is measured in each of three calibration standards and the mean RT is calculated as the average of the three values. An RT is measured for the surrogates in each of the three analytes of Individual Mixture A during the initial calibration and the mean RT is calculated as the average of the three values.

8.3 A retention time window is calculated for each single component analyte and surrogate by using the list in paragraph 8.4. Windows are centered around the mean absolute retention time for the analyte established during the initial calibrations.

8.4 Retention time windows for single and multicomponent analytes and surrogates.

Compound	Retention Time Window in Minutes
alpha-BHC	± 0.05
beta-BHC	± 0.05
gamma-BHC	± 0.05
delta-BHC	± 0.05
Heptachlor	± 0.05
alpha-Chlordane	± 0.07
gamma-Chlordane	± 0.07
Aldrin	± 0.05
Heptachlor epoxide	± 0.07
Dieldrin	± 0.07
Endrin	± 0.07
Endrin aldehyde	± 0.07
Endrin ketone	± 0.07
DDD	± 0.07
DDE	± 0.07
DDT	± 0.07
Endosulfan I	± 0.07
Endosulfan II	± 0.07

Endosulfan sulfate	±	0.07
Methoxychlor	±	0.07
Aroclors	±	0.07
Toxaphene	±	0.07
Tetrachloro-m-xylene	±	0.05
Decachlorobiphenyl	±	0.10

8.5 For each multicomponent analyte, the RTs for three to five peaks are calculated from the initial calibration standard analysis. An RT window of +0.07 minutes is used for all multi-component analyte peaks.

8.6 Analytes are identified when peaks are observed in the RT window for the compound on both GC columns.

APPENDIX VI

Calibration Factors and Criteria

This Appendix references Section III D 9.2.4 of USEPA CLP OLM03.1 SOW.

9. Calibration Factors for Single Pesticides

9.1 During the initial calibration sequence, the Contractor must establish the magnitude of the linear ECD response range for each single component pesticide and surrogate on each column and for each GC system. This is accomplished by analyzing the Individual Standard Mixtures A and B at three concentrations during the initial calibration sequence.

9.2 The linearity of the instrument is determined by calculating a percent relative standard deviation (%RSD) of the calibration factors from a three-point calibration curve for each single component pesticide and surrogate. Either peak area or peak height may be used to calculate calibration factors used in the %RSD equation. For example, it is permitted to calculate linearity for Endrin based on peak area and to calculate linearity for Aldrin based on peak height. It is not permitted within a %RSD calculation for an analyte to use calibration factors calculated from both peak area and peak height. For example, it is not permitted to calculate the calibration factor for the low point standard for endrin using peak height and calculate the midpoint and high point standard calibration factors for endrin using peak area.

9.2.1 Calculate the calibration factor for each single component pesticide and surrogate over the initial calibration range using Equation 5.

9.2.2 Calculate the mean and the %RSD of the calibration factors for each single component pesticide and surrogate over the initial calibration range using Equations 6 and 7.

$$CF = \frac{\text{Peak Area (or Height) of the Standard}}{\text{Mass Injected (ng)}} \qquad \text{EQ. 5}$$

$$\overline{CF} = \sum_{i=1}^{n} \frac{CF_i}{n} \qquad \text{EQ. 6}$$

$$\%RSD = \frac{SD}{\overline{CF}} \times 100 \qquad \text{EQ. 7}$$

Where: $$SD = \frac{\sum_{i=1}^{n}\left(CF_i - \overline{CF}\right)^2}{n-1}$$ and n=3

9.2.3 The linearity of the calibration is considered acceptable when the %RSD of the three point calibration is less than 20.0 percent (alpha-BHC and delta-BHC less than 25.0 percent) except noted in the following.

The % RSD of the two surrogates must be less than or equal to 30.0 percent. Up to two single compound target compounds (but not surrogates) per column may be exceed the 20.0 percent limit for % RSD. (25.0 percent for alpha-BHC and delta-BHC), but those compounds must have a % RSD of less than or equal to 30.0 percent.

9.2.4 If the linearity requirements listed above are met, the calibration factor from the mid point concentration standard is used for quantitation of each single component pesticide.

9.3 Sample analysis may not proceed until a satisfactory calibration has been demonstrated.

10. Calibration Factors for Toxaphene and Aroclors

10.1 Toxaphene and Aroclors require only a single-point calibration and they present special analytical difficulties. Because of the alteration of these materials in the environment, it is probable that samples which contain multicomponent analytes will give patterns similar to, but not identical with, those of the standards.

10.2 A set of three to five major peaks is selected for each multicomponent analyte. Retention times and calibration factors are determined from the initial calibration analysis for each peak.

11. Acceptance Criteria for Chromatograms of Calibration Standards

The identification of single component pesticides by gas chromatographic methods is based primarily on retention time data. The retention time of the apex of a peak can be verified only from an on-scale chromatogram. The identification of multicomponent analytes is based primarily on recognition of patterns of retention times displayed on a chromatogram. Therefore, the following requirements apply to all data presented for single component and multicomponent analytes.

11.1 The chromatograms that result from the analyses of the Resolution Check Mixture, the Performance Evaluation Mixture, and Individual Standard Mixtures A and B during the

initial calibration sequence must display the single component analytes present in each standard at greater than 10 percent of full scale but less than 100 percent of full scale.

11.2 The chromatograms, for at least one of the three analyses each of Individual Standard Mixtures A and B from the initial calibration sequence, must display the single component analytes at greater than 50 percent and less than 100 percent of full scale.

11.3 The chromatograms of the standards for the multicomponent analytes analyzed during the initial calibration sequence must display the peaks chosen for identification of each analyte at greater than 25 percent and less than 100 percent of full scale.

11.4 For any standard containing alpha-BHC, the baseline of the chromatogram must return to below 50 percent of full scale before the elution time of alpha-BHC, and return to below 25 percent of full scale after the elution time of alpha-BHC and before the elution time of decachlorobiphenyl.

11.5 If a chromatogram is replotted electronically to meet requirements, the scaling factor used must be displayed on the chromatogram.

11.6 If the chromatogram of any standard needs to be replotted electronically to meet these requirements, both the initial chromatogram and the replotted chromatogram must to submitted in the data package.

APPENDIX VII

Sample Analysis

This Appendix references to Section III D 10.2 of USEPA CLP OLM03.1 SOW.

1. Sample Analysis

1.1 Unless ambient temperature on-column injection is used (see paragraph 4.2), the injector must be heated to at least 200°C. The gas chromatographic conditions from paragraph 4 must be used.

1.2 The injection must be made on-column by using either automatic or manual injection. If autoinjectors are used, 1.0 µL injection volumes may be used. Manual injections shall use at least 2.0 µL injection volumes. The same injection volume must be used for all standards, samples, and blanks associated with the same initial calibration. If a single injection is used for two GC columns attached to a single injection port, it may be necessary to use an injection volume greater than 2 µL. However, the same injection volume must be used for all analyses.

1.3 Analysis of a sample on both GC columns is required for all samples, blanks, matrix spikes, and matrix spike duplicates.

1.4 The requirements for the analysis sequence apply to both GC columns and to all instruments used for these analyses.

1.5 The laboratory will identify and quantitate analyte peaks based on RT and calibration factor established during the initial calibration sequence, as long as an acceptable calibration verification (see Appendix IV) is performed every 12 hours.

1.6 The protocol is intended to achieve the quantitation limits shown in Exhibit C whenever possible. If sample chromatograms have interfering peaks, a high baseline, or off-scale peaks, then those samples must be reanalyzed following dilution, further cleanup, or re-extraction. Samples which cannot be made to meet the given specifications after one re-extraction and three-step cleanup (GPC, Florisil, and sulfur removal) are reported in the SDG Narrative and do not require further analysis. No limit is placed on the number of re-extractions of samples that may be required because of contaminated method blanks.

1.7 The sample must be analyzed at the most concentrated level that is consistent with achieving satisfactory chromatography (defined below). If dilution is employed solely to bring a peak within the calibration range or to get a multicomponent pattern on scale, the results for both

the more and the less concentrated extract must be reported. The resulting changes in quantitation limits and surrogate recovery must be reported also for the diluted samples.

1.8 If the Contractor has reason to believe that diluting the final extracts will be necessary, an undiluted run may not be required. If an acceptable chromatogram (as defined below) is achieved with the diluted extract, an additional extract 10 times the concentration of the dilute sample must be injected and reported with the sample data.

1.9 No target analyte concentrations may exceed the upper limit of the initial calibration.

1.10 A standard for any identified multicomponent analyte must be analyzed on the same instrument within 72 hours of its detection in a sample.

1.11 The identification of single component pesticides by gas chromatographic methods is based primarily on retention time data. The retention time of the apex of a peak can be verified only from an on-scale chromatogram. The identification of multicomponent analytes is based primarily on recognition of patterns of retention times displayed on a chromatogram. Therefore, the following requirements apply to all data presented for single component and multicomponent analytes.

1.11.1 When no analytes are identified in a sample, the chromatograms from the analyses of the sample extract must use the same scaling factor as was used for the low point standard of the initial calibration associated with those analyses.

1.11.2 Chromatograms must display single component pesticides detected in the sample at less than full scale.

1.11.3 Chromatograms must display the largest peak of any multicomponent analyte detected in the sample at less than full scale.

1.11.4 If an extract must be diluted, chromatograms must display single component pesticides between 10 and 100 percent of full scale.

1.11.5 If an extract must be diluted, chromatograms must display the peaks chosen for quantitation of multicomponent analytes between 25 and 100 percent of full scale.

1.11.6 For any sample, the baseline of the chromatogram must return to below 50 percent of full scale before the elution time of alpha-BHC, and return to below 25 percent of full scale after the elution time of alpha-BHC and before the elution time of decachlorobiphenyl.

1.11.7 If a chromatogram is replotted electronically to meet these requirements, the scaling factor used must be displayed on the chromatogram.

1.11.8 If the chromatogram of any sample needs to be replotted electronically to meet these requirements, both the initial chromatogram and the replotted chromatogram must be submitted in the data package.

2. Quantitation of Analytes

2.1 Quantitation must be performed and reported on both columns.

2.2 Analytes must be quantitated with an electronic integrator or with a laboratory data system. The analyst can use either peak height or peak area as the basis for quantitation. The use of an electronic integrator or a laboratory data system is required.

2.3 The chromatograms of all samples must be reviewed by a qualified pesticide analyst before they are reported.

2.4 In order to be quantitated, the detector response (peak area or peak height) of all of the single component analytes must lie between the response of the low and high concentrations in the initial calibration. If the analytes are detected below the CRQL, they are reported as present below the CRQL, and flagged according to the instructions in exhibit B. If they are detected at a level greater than the high calibration point, the sample must be diluted either to a maximum of 1:100,000 or until the response is within the linear range established during calibration. Guidance in performing dilutions and exceptions to this requirement are given below.

2.4.1 If the response is still above the high calibration point after the dilution of 1:100,000, contact the client immediately.

2.4.2 Use the results of the original analysis to determine the approximate dilution factor required to get the largest analyte peak within the initial calibration range.

2.4.3 The dilution factor chosen should keep the response of the largest peak for a target compound in the upper half of the initial calibration range of the instrument.

2.4.4 Do not submit data for more than two analyses, i.e., the original sample extract and one dilution, or, if a screening procedure was employed, from the most concentrated dilution analyzed and one further dilution.

2.4.5 Do not dilute MS/MSD samples to get either spiked or non-spiked analytes within the calibration range. If the sample from which the MS/MSD aliquots were taken contains high levels of the spiked analytes, calculate the concentration and recovery of the analytes from the undiluted analysis and note the problem in the SDG Narrative.

2.5 The concentrations of the single component pesticides are calculated by using the following equations:

2.5.1 Water

$$\text{Concentration} \quad ug/L = \frac{(Ax)\,(Vt)\,(Df)(GPC)}{(CF)\,(Vo)\,(Vi)} \qquad \text{EQ. 8}$$

Where

Ax = Area of the peak for the compound to be measured

CF = Calibration factor for the mid point concentration external standard (area per ng)

Vo = Volume of water extracted in milliliters (mL)

Vi = Volume of extract injected in microliters (uL)

Vt= Volume of the concentrated extract in microliters (uL)

GPC = GPC factor. (If no GPC is performed, GPC = 1. If GPC is performed, then GPC = 2).

Df = Dilution Factor. The dilution factor for analysis of water samples by this method is defined as follows:

$$\frac{\text{uL most conc. extract used to make dilution + uL clean solvent}}{\text{uL most conc. extract used to make dilution}}$$

If no dilution is performed, Df = 1.0.

2.5.2 Soil/Sediment (assuming GPC Clean-up is used)

Concentration (Dry weight basis) ug/Kg $= \frac{(Ax)\,(Vt)\,(Df)\,(GPC)}{(CF)\,(Vi)\,(Ws)\,(D)}$ EQ. 9

Where:

Ax and CF are as given for water, above.

Vt = Volume of the concentrated extract in microliters (µL) *(This volume must be 5000 µL)*

Vi = Volume of extract injected in microliters (µL)

D $= \frac{100 - \%\ \text{moisture}}{100}$

Ws = Weight of sample extracted in grams (g)

GPC = GPC factor. (If no GPC is performed, GPC = 1. If GPC is performed, then GPC = 2). Note that GPC clean-up is required for all soil sample extracts.

Df = Dilution Factor. The dilution factor for analysis of soil samples by this method is defined as follows:

$$\frac{\text{uL most conc. extract used to make dilution} + \text{uL clean solvent}}{\text{uL most conc. extract used to make dilution}}$$

If no dilution is performed, Df = 1.0.

The factor of 2.0 in the numerator is used to account for the amount of extract that is not recovered from the mandatory use of GPC cleanup. Concentrating the extract collected after GPC to 5.0 mL rather than 10.0 mL for water samples not subjected to GPC, maintains the sensitivity of the soil method comparable to that of the water method, but correction of the numerical result is still required.

2.5.3 Note that the calibration factors used for the quantitation of the single component pesticides are the calibration factors from the mid point concentration standard for each analyte.

2.5.4 Because of the likelihood that compounds co-eluting with the target compounds will cause positive interferences and increase the concentration determined by the method, the lower of the two concentrations calculated for each single component pesticide is reported on Form I.

In addition, the concentrations calculated for both the GC columns are reported on Form X, along with a percent difference comparing the two concentrations. The percent difference is calculated according to Equation 10 below.

EQ. 10

$$\%D = \frac{Conc._H - Conc._L}{Conc._L} \times 100$$

Where:

Conc.H = The higher of the two concentrations for the target compound in question

Conc.L = The lower of the two concentrations for the target compound in question

Note that using this equation will result in percent difference values that are always positive. The value will also be greater than a value calculated using the higher concentration in the denominator. However, given the likelihood of a positive interference raising the concentration determined on one GC column, this is a conservative approach to comparing the two concentrations.

2.6 The concentrations of the surrogates are calculated in a similar manner as the other analytes, using Equation 8. and 9. The recoveries of the surrogates are calculated according to Equation 11.

EQ. 11

$$SurrogatePercent\,Recovery = \frac{Qd}{Qa} \times 100$$

Qd = Quantity determined by analysis

Qa = Quantity added to sample/blank

The limits for the recovery of the surrogates are 30-150 percent for both surrogate compounds. As these limits are only advisory, no further action is required by the laboratory. However, frequent failures to meet the limits for surrogate recovery warrant investigation by the laboratory.

2.7 The quantitative determination of Toxaphene or Aroclors is somewhat different from that of single component analytes. Quantitation of peaks within the detector linear range CRQL to > 16 times CRQL is based on a single calibration point assuming linear detector response.

Alternatively, a linear calibration range may be established during a run sequence by a three-point calibration curve for any multicomponent analyte.

2.8 The reporting requirements for multicomponent analytes are similar to those for single component analytes. If the concentration is calculated to be 10^6 times the CRQL, contact the client immediately.

2.9 The quantitation of toxaphene or Aroclors must be accomplished by comparing the heights or the areas of each of the three to five major peaks of the multicomponent analyte in the sample with the calibration factor for the same peaks established during the initial calibration sequence. The concentration of multicomponent analytes is calculated by using Equations 8 and 9, where Ax is the area for each of the major peaks of the multicomponent analyte. The concentration of each peak is determined and then an average concentration for three to five major peaks is determined and reported on Form I (Exhibit B). The following table lists the number of potential quantitation peaks for each Aroclor and Toxaphene.

Analyte	No. of Potential Quantitation Peaks
Aroclor 1016/1260	5
Aroclor 1221	3
Aroclor 1232	4
Aroclor 1242	5
Aroclor 1248	5
Aroclor 1254	5
Toxaphene	4

2.10 The choice of the peaks used for multicomponent quantitation and the recognition of those peaks may be complicated by the environmental alteration of the Toxaphene or Aroclors, and by the presence of coeluting analytes or matrix interferences, or both.

2.11 If more than one multicomponent analyte is observed in a sample, the Contractor must choose separate peaks to quantitate the different multicomponent analytes. A peak common to both analytes present in the sample must not be used to quantitate either compound.

2.12 The reporting requirements for Toxaphene and the Aroclors are similar to those for the single component analytes, except that the lower mean concentration (from the three to five peaks) is reported on the Form I, and the two mean concentrations reported on the Form X. The two mean concentrations are compared by calculating the percent difference using equation 10.

3. Sample Data Acceptance Criteria

3.1 The requirements above apply to both columns, and quantitation must be performed on both GC columns and reported.

3.2 All samples must be analyzed as part of a valid analysis sequence (paragraph 5). They must be bracketed by acceptable instrument blanks (paragraph 15.3), acceptable Performance Evaluation Mixtures, and acceptable Individual Standard Mixtures A and B (appendix IV) that were analyzed at the required frequency.

3.3 The retention times for both of the surrogates must be within the retention time windows as calculated in paragraph 8.

3.4 Reportable data for a sample must include a chromatogram in which a baseline returns to below 50 percent of full scale before the elution time of alpha-BHC, and to below 25 percent of full scale after alpha-BHC and before decachlorobiphenyl.

3.5 If dilution has been applied and if no peaks are detected above 25 percent of full scale, analysis of a more concentrated sample is required.

3.6 Reportable sample data must include chromatogram(s) which meet the criteria in paragraph 12.11.

4. Blanks

There are two types of blanks required by this method: the method blank and the instrument blank. A separate sulfur cleanup blank may be required if all samples associated with a given method blank are not subjected to sulfur cleanup. Samples that are associated with a sulfur cleanup blank are also associated with the method blank with which they were extracted. Both the method and sulfur cleanup blanks must meet the respective acceptance criteria for the sample analysis acceptance criteria to be met.

4.1 Method blank

4.1.1 Method blanks are spiked with the surrogate solution, extracted, cleaned up, and analyzed by following the same procedure that is used with the samples. A water method blank is one liter of reagent water treated as the water sample aliquot. A soil method blank is 30 g of sodium sulfate treated as the soil sample aliquot.

Method blank analysis must be performed once for the following, whichever is most frequent, and analyzed on each GC/EC system used to analyze samples:

- Each SDG (not to exceed 20 field samples), or
- Each matrix within an SDG, or
- Each extraction procedure within an SDG, or
- Whenever samples are extracted.

4.1.2 In order to be acceptable, a method blank analysis cannot contain any of the analytes listed in Exhibit C at greater than the CRQL. The surrogate retention times must be within the retention time windows calculated from the initial calibration sequence mean retention time for both tetrachloro-m-xylene and decachlorobiphenyl. The surrogate recoveries must fall within the acceptance windows of 30-150%. In the case of a method blank, these limits are not advisory.

4.1.3 All samples associated with an unacceptable method blank (see Form IV) must be re-extracted and reanalyzed.

4.2 Sulfur Cleanup Blank.

4.2.1 The sulfur cleanup blank is a modified form of the method blank. The sulfur cleanup blank is hexane spiked with the surrogates and passed through the sulfur cleanup procedure (see Section II, paragraph 7.4).

4.2.2 The sulfur cleanup blank is prepared when only part of a set of samples extracted together requires sulfur removal. A method blank is associated with the entire set of samples. The sulfur cleanup blank is associated with the part of the set which required sulfur cleanup. If all the samples associated with a given method blank are subjected to sulfur cleanup, then the method blank must be subjected to sulfur cleanup, and no separate sulfur cleanup blank is required.

4.2.3 In order to be acceptable, a sulfur blank analysis cannot contain any of the analytes listed in Exhibit C at greater than the CRQL. The surrogate retention times must be within the retention time windows calculated from the initial calibration sequence mean retention time for both tetrachloro-m-xylene and decachlorobiphenyl and surrogate recoveries must be within the acceptance windows of 30-150%. In the case of a sulfur clean-up blank, the limits are not advisory.

4.2.4 All samples associated with an unacceptable sulfur blank (see Form IV) must be re-extracted and reanalyzed.

4.3 Instrument blank

4.3.1 An instrument blank is a hexane or iso-octane solution containing 20.0 ng/mL of tetrachloro-m-xylene and decachlorobiphenyl and 50.0 ng/mL of isodrin.

4.3.2 The first analysis in a 12-hour analysis sequence must be an instrument blank. All acceptable samples analyses are to be bracketed by acceptable instrument blanks, as described in paragraph 5.1.

4.3.3 An acceptable instrument blank must be analyzed within a 12-hour analysis sequence and must demonstrate that no analyte in Exhibit C is detected at greater than 0.5 times the CRQL and that the surrogate retention times are within the retention time windows.

4.3.4 If analytes are detected at greater than half the CRQL or the surrogate RTs are outside the RT windows, all data collection must be stopped, and corrective action must be taken. Data for samples which were run between the last acceptable instrument blank and the unacceptable blank are considered suspect. An acceptable instrument blank must be run before additional data are collected. After an acceptable instrument blank is run, all samples which were run after the last unacceptable instrument blank must be reinjected during a valid run sequence and must be reported.

4.3.5 Analysts are cautioned that running an instrument blank once every 12 hours is the minimum contract requirement. Late eluting peaks may carry over from one injection to the next if highly complex samples are analyzed or if the GC conditions are unstable. Such carryover is unacceptable. Therefore, it may be necessary to run instrument blanks more often to avoid discarding data.

APPENDIX VIII

MS/MSD

This Section references Section III D 12.2 of USEPA CLP OLM03.1 SOW.

1. Matrix Spike/Matrix Spike Duplicate

1.1 A matrix spike and matrix spike duplicate must be extracted and analyzed at least once with every 20 samples of each matrix. NOTE: There is no differentiation between "low" and "medium" soil samples in this method. Therefore only one soil MS/MSD is to be submitted.

1.2 The surrogate retention times must be within the retention time windows specified.

1.3 The percent recoveries and the relative percent difference between the recoveries of each of the 6 compounds in the matrix spike samples will be calculated and reported by using the following equations:

$$\text{Matrix Spike Recovery} = \frac{SSR - SR}{SA} \times 100 \qquad \text{EQ. 12}$$

Where

SSR = Spike sample result
SR = Sample result
SA = Spike added

$$RPD = \frac{|MSR - MSDR|}{\frac{1}{2}(MSR + MSDR)} \times 100 \qquad \text{EQ. 13}$$

Where

RPD = Relative percent difference
MSR = Matrix spike recovery
MSDR = Matrix spike duplicate recovery

The vertical bars in the formula above indicate the absolute value of the difference, hence RPD is always expressed as a positive value.

1.4 The Contractor shall report matrix spike and matrix spike duplicate recoveries and percent difference values with the analytical results (see Exhibit B). The limits for matrix spike compound recovery and RPD are given below. As these limits are only advisory, no further action by the laboratory is required, however, frequent failures to meet the limits for recovery or RPD warrant investigation by the laboratory.

MATRIX SPIKE RECOVERY AND
RELATIVE PERCENT DIFFERENCE LIMITS

Compound	% R Water	RPD Water	% R Soil	RPD Soil
gamma-BHC (Lindane)	56-123	15	46-127	50
Heptachlor	40-131	20	35-130	31
Aldrin	40-120	22	34-132	43
Dieldrin	52-126	18	31-134	38
Endrin	56-121	21	42-139	45
4,4'-DDT	38-127	27	23-134	50

APPENDIX IX

Form Instructions

This Appendix references Section III B of the USEPA CLP OLM03.1 SOW.

SECTION III B

FORM INSTRUCTION GUIDE

This section includes specific instructions for the completion of all required forms. Each of the forms is specific to a given fraction (volatile, semivolatile, pesticide/Aroclor), and in some instances specific to a given matrix (water or soil) within each fraction. The contractor shall submit only those forms pertaining to the fractions analyzed for a given sample or samples. For instance, if a sample is scheduled for volatile analysis only, provide only VOA forms. There are two pages relating to the semivolatile fraction for Forms I, VI, VII, and VIII. whenever semivolatiles are analyzed and one of the above-named forms is required, both pages (SV-1 and SV-2) must be submitted. These instructions are arranged in the following order:

A. General Information and Header Information

B. Organic Analysis Data Sheets (Form I)

C Surrogate Recovery (Form II PEST)

D. Matrix Spike/Matrix Spike Duplicate Recovery (Form III)

E. Method Blank Summary (Form IV)

F. GC Initial Calibration Data (From VI PEST-1, PEST-2, PEST-3, PEST-4)

G. GC/EC Continuing Calibration (Form VII PEST)

H. Pesticide Analytical Sequence (Form VIII PEST)

I. Pesticide Cleanup Procedures (Form IX PEST-1, PEST-2)

J. Pesticide/Aroclor Identification (Form X PEST-1, PEST-2)

K. Sample Log-In Sheet (Form DC-1)

L. Document Inventory Sheet (Form DC-2)

A. General Information and Header Information

A.1. The data reporting forms presented in Section IV have been designed in conjunction with the computer-readable data format specified in Exhibit H, Data Dictionary and Format for Data Deliverables in Computer-Readable Format. The specific length of each variable for computer-readable data transmission purposes is given in the Data Dictionary (Exhibit H). Information entered on these forms must not exceed the size of the field given on the form, including such laboratory-generated items as Lab Name and Lab Sample ID.

A.2. Note that on the hard copy forms (Section IV), the space provided for entries is greater in some instances than the length prescribed for the variable as written to diskette (see Exhibit H). Greater space is provided on the hard copy forms for the sake of visual clarity.

A.3. Values must be reported on the hard copy forms according to the individual form instructions in this Section. For example, results for concentrations of VOA target compounds must be reported to two significant figures if the value is greater than or equal to 10. Values can be written to the diskette file in any format that does not exceed the field specification as given in the record specifications and discussed in "Record Structure", paragraph 5 of Exhibit H.

A.4. All characters which appear on the data reporting forms presented in the contract (Exhibit B, Section IV) must be reproduced by the Contractor when submitting data, and the format of the forms submitted must be identical to that shown in the contract. No information may be added, deleted, or moved from its specified position without prior written approval of the EPA Administrative Project Officer. The names of the various fields and compounds (i.e., "Lab Code," "Chloromethane") must appear as they do on the forms in the contract, including the options specified in the form (i.e., "Matrix: (soil/water)" must appear, not just "Matrix"). For items appearing on the uncompleted forms (Section IV), the use of uppercase and lowercase letters is optional.

A.5. Alphabetic entries made onto the forms by the Contractor shall be in ALL UPPERCASE letters (i.e., "LOW", not "Low" or "low"). If an entry does not fill the entire blank space provided on the form, null characters shall be used to remove the remaining underscores that comprise the blank line. See Exhibit H for more detailed instructions. However, do not remove the underscores or vertical bar characters that delineate "boxes" on the forms. The only exception would be those underscores at the bottom of a "box" that are intended as a data entry line (for instance, on Form 2A, line 30, if data must be entered on line 30, it will replace the underscores).

A.6. Six pieces of information are common to the header sections of each data reporting form. They are Lab Name, Contract, Lab Code, Case No., SAS No., and SDG No. Except as noted below for SAS No., this information must be entered on every form and must match on every form.

A.7. The "Lab Name,, shall be the name chosen by the Contractor to identify the laboratory. It may not exceed 25 characters.

A.8. The "Lab Code" is an alphabetical abbreviation of up to 6 letters, assigned by the EPA, to identify the laboratory and aid in data processing. This lab code shall be assigned by the EPA at the time a contract is awarded, and shall not be modified by the Contractor, except at the direction of the EPA. If a change of name or ownership occurs at the laboratory, the lab code will remain the same until the contractor is directed by the EPA to use another lab code assigned by the EPA.

A.9. The "Case No." is the EPA-assigned Case number associated with the sample, and reported on the Traffic Report.

A.10. The "Contract" is the number of the EPA contract under which the analyses were performed. In the case of multiple laboratories operating under a corporate-wide contract, the contract number entered shall be that of the corporate contract, regardless of the facility performing the analyses (see Lab Code, above).

A.11. The IISDG No." is the Sample Delivery Group number. The Sample Delivery Group (SDG) number is the EPA Sample Number of the first sample received in the SDG. When several samples are received together in the first SDG shipment, the SDG number shall be the lowest sample number (considering both alpha and numeric designations) in the first group of samples received under the SDG.

A.12. The "SAS No." is the EPA-assigned number for analyses performed under Special Analytical Services. If samples are to be analyzed under SAS only and reported on these forms, then enter SAS No. and leave Case No. blank. If samples are analyzed according to the "Routine Analytical Services" (IFB) protocols and have additional "SAS" requirements, list both Case No. and SAS No. on all forms. If the analyses have no SAS requirements, leave "SAS No." blank. NOTE: Some samples in an SDG may have a SAS No. while others do not.

A.13. The other information common to most of the forms is the "EPA Sample No.". This number appears either in the upper right-hand corner of the form, or as the left column of a table summarizing data from a number of samples. When the "EPA Sample No." is entered into the triple-spaced box in the upper right-hand corner of Form I, Form IV, or Form X, it should be entered on the middle line of the three lines that comprise the box.

A.14. All samples, matrix spikes, matrix spike duplicates, blanks, and standards shall be identified with an EPA Sample Number. For field samples, matrix spikes and matrix spike duplicates, the EPA Sample Number is the unique identifying number given in the Traffic Report that accompanied that sample.

A.15. In order to facilitate data assessment, the following sample suffixes must be used:

XXXXX	=	EPA sample number
XXXXXMS	=	Matrix spike sample
XXXXXMSD	=	Matrix spike duplicate sample
XXXXXRE	=	Re-extracted and re-analyzed sample
XXXXXDL	=	Sample analyzed at a secondary dilution

A.16. Form VIII Pest requires that all samples analyzed in a given analytical sequence be listed, regardless of whether or not they are part of the SDG being reported. Therefore, use "ZZZZZ" as the EPA Sample No. for any sample analysis not associated with the SDG being reported.

A.17. For blanks and standards, the following identification scheme must be used as the "EPA Sample No."

1. Volatile banks shall be identified as VBLK##.

2 Semivolatile blanks shall be identified as SBLK##.

3. Pesticide/Aroclor method blanks shall be identified as PBLK##.

4. Pesticide/Aroclor instrument blanks shall be identified as PIBLK##.

A.18. The "EPA Sample No." must be unique for each blank within an SDG. Within a fraction, a laboratory must achieve this be replacing the two-character "##" terminator of the identifier with one or two characters or numbers, or a combination of both. For example, possible, identifiers for volatile blanks would be VBLK1, VBLK2, VBLKA1, VBLKB2, VBLK10, VELKAB, etc.

A.19. Volatile and semivolatile standards shall be identified as SFTD###, were

F = Fraction (V for volatiles; S for semivolatiles).

STD = Indicates a standard.

= The concentration in ug/L of volatile standards (i.e., 010, 020, 050, 100, and 200) or the amount injected in ng for semivolatile standards (i.e., 020, 050, 080, 120, and 160).

A.20. As for the blank identifiers, these designations will have to be concatenated with other information to uniquely identify each standard.

A.21. For pesticide/Aroclor standards, the following scheme shall be used to enter "EPA Sample Number".

Name		EPA Sample Number
Individual Mix A	(low point)	INDAL##
Individual Mix A	(mid point)	INDAM##
individual Mix A	(high point)	INDAH##
Individual Mix B	(low point)	INDBL##
Individual Mix B	(mid point)	INDBM##
Individual Mix B	(high point)	INDBH##
Resolution Check		RESC##
Performance Evaluation Mixture		PEM##
Toxaphene		TOXAPH##
Aroclor 1016		AR1016##
Aroclor 1221		AR1221##
Aroclor 1232		AR1232##
Aroclor 1242		AR1242##
Aroclor 1248		AR1248##
Aroclor 1254		AR1254##
Aroclor 1260		AR1260##

A.22. The permitted mixture of Aroclor 1016 and Aroclor 1260 shall be entered as AR1660##.

A.23. If the standards are injected onto both GC columns on the same instrument simultaneously, the same EPA Sample Number may be used for reporting data for the standards for both columns. If simultaneous injections are not made, then the same number may not be used.

A.24. Several other pieces of information are common to many of the Data Reporting Forms. These include Matrix, Sample wt/vol., Level, Lab Sample ID, and Lab File ID.

A.25. For "Matrix", enter "SOIL" for soil/sediment samples, and enter "WATER" for water samples. NOTE: The matrix must be spelled out. Abbreviations such as "S" or "W" shall not be used.

A.26. For "Sample wt/vol." enter the number of grams (for soil) or milliliters (for water) of sample used in the first blank line, and the units, either "G" or "ML", in the second blank.

A.27. For Pesticide/Aroclor forms, there is no differentiation between low and medium soil samples and no level is entered on any of these forms.

A.28. "Lab Sample ID" is an optional laboratory-generated internal identifier. Up to 12 alpha-numeric characters may be reported here. If the contractor does not have a Lab Sample ID, this field may be left blank.

A.29. "Lab File ID" is the laboratory-generated name of the GC/MS data system file containing information pertaining to a particular analysis. Up to 14 alpha-numeric characters may be used here.

A.30. "Instrument ID" is common to many of the forms, particularly those containing calibration data. The identifier used by the laboratory must include some indication of the manufacturer and/or model of the instrument, and contain additional characters that differentiate between all instrument of the same type in the laboratory.

A.31. "GC Column" and "ID (mm)" are common to various other forms. These two fields are to be used to identify the stationary phase of the GC column (previously called GC Column ID), and the internal diameter of the GC column in millimeters (mm). For packed columns, convert the ID from inches to millimeters as necessary, and enter in the "ID" field.

A.32. Forms II, IV, V, VIII, IX, and X contain a field labeled "page _ of _" in the bottom left-hand corner. If the number of entries required on any of these forms exceeds the available space, continue entries on another copy of the same fraction-specific form duplicating all header information. if a second page is required, number the pages consecutively, as "page 1 of 2" and "page 2 of 2." If a second page is not required, number the page "page 1 of 1." NOTE: These forms are fraction-specific, and often matrix-specific within fraction. For example, Form II VOA-1 and Form II VOA-2 are for different data. Therefore, do not number the pages of all six versions of Form II as "1 of 6, 2 of 6, etc." Number only pages within a fraction-specific and matrix specific form.

A.33. For rounding off numbers to the appropriate level of precision, observe the following common rules. If the figure following those to be retained is less than 5, drop it (round down). if the figure is greater than 5, drop it and increase the last digit to be retained by 1 (round up). If the figure following the last digit to be retained equals 5, round up if the digit to be retained is odd, and round down if that digit is even.

B. Organic Analysis Data Sheet (Form I)

B.1. Form I PEST

B.1.1. This form is used for tabulating and reporting sample analysis results for target compounds. If all fractions are not requested to be analyzed, only the pages specifically required must be submitted. If the pesticide/Aroclor analysis is the only analysis requested, only Form I Pest must be submitted for that sample.

B.1.2. Complete the header information on each page of Form I required, according to the instructions in Part A and as follows:

B.1.3. For pesticides/Aroclors, enter the values for the percent moisture determined during the analysis. In the field "decanted (Y/N)", enter "Y" if the sample had standing water above the soil/sediment that was decanted, or "N" if no water was decanted off the surface of the sample. Report percent moisture (decanted or not decanted) to the nearest whole percentage point (i.e., 5%, not 5.3%). Leave these fields blank for Form I for method blanks and instrument blanks.

B.1.4. For pesticides/Aroclors, enter the method of extraction as "SEPF" for separatory funnel, "CONT" for continuous liquid-liquid extraction, or "SONC" for sonication (soils only).

B.1.5. If gel permeation chromatography, "GPC Cleanup", was performed, enter "Y" for yes. Otherwise, enter "N" for no, if GPC was not performed. NOTE: GPC is required for all soil samples analyzed for semivolatiles and pesticides/Aroclors, therefore all soil sample forms will contain "Y" in this field.

B.1.6. For soil samples only, enter pH for semivolatiles and pesticides/Aroclors, reported to 0.1 pH units.

B.1.7. "Date Received" is the date of sample receipt at the laboratory, as noted on the Traffic Report (i.e., the VTSR). It should be entered as MM/DD/YY.

B.1.8. "Date Extracted" and "Date Analyzed" should be entered in a similar fashion. If continuous liquid-liquid extraction procedures are used, enter the date on which the procedure was started for "Date Extracted". If separatory funnel or sonication procedures are used, enter the data on which the procedure was completed. For pesticide/Aroclor samples, the date of analysis should be the date of the first GC analysis performed. The date of sample receipt will be compared with the extraction and analysis dates of each fraction to ensure that contract holding times were not exceeded.

B.1.9. For pesticides/Aroclors, enter the actual volume of the most concentrated sample extract, in microliters, under "Concentrated Extract Volume". This volume typically will be 1000 µL, or 500 µL when GPC was performed. If a dilution of the sample extract is made in a subsequent analysis, this volume will remain the same, but the dilution factor will change.

B.1.10. For pesticides/Aroclors, enter the volume of the sample extract injected into the GC under "Injection Volume". Report this volume in microliters to one decimal place, i.e., 1.0 µL. Note: A 2.0 microliter injection is required for semivolatile analyses.

B.1.11. If a sample or sample extract has been diluted for analysis, enter the "Dilution Factor" as a single number, not a fraction, such as "100.0," for a 1 to 100 dilution of the sample. Enter 0.1 for a concentration of 10 to 1. If a sample was not diluted, enter "1.0." Reported dilution factors to one decimal place.

B.1.12. For positively identified target compounds, the Contractor shall report the concentrations detected as uncorrected for blank contaminants.

B.1.13. Report all pesticide/Aroclor results to two significant figures.

B.1.14. The appropriate concentration units, µg/L or µg/kg, must be entered.

B.1.15. If the result is a value greater than or equal to the quantitation limit, report the value.

B 1.16. Under the column labeled "Q" for qualifier, flag each result with the specific Data Reporting Qualifiers listed below. The Contractor is encouraged to use additional flags or footnotes. The definition of such flags must be explicit and must be included in the SDG Narrative.

B.1.17. For reporting results, the following contract specific qualifiers are to be used. The seven qualifiers defined below are not subject to modification by the laboratory. Up to five qualifiers may be reported on Form I for each compound.

B.1.18. The seven EPA-defined qualifiers to be used are as follows:

U - Indicates compound was analyzed for but not detected. the sample quantitation limit must be corrected for dilution and for percent moisture. For example, 10 U for phenol in water if the sample final volume is the protocol-specified final volume. If a 1 to 10 dilution of extract is necessary, the reported limit is 100 U. For a soil sample, the value must also be adjusted for percent moisture. For example, if the sample had 24% moisture and a 1 to 10 dilution factor, the sample quantitation limit for phenol (330 U) would be corrected to

$$\frac{(330\ U)}{D} \times df$$

Where

$$D = \frac{100 - \%\ moisture}{100}$$

and Df = dilution factor

For example, at 24% moisture, $D = \frac{100 - 24}{100} = 0.76$

(330 U) x 10 = 4300. U (rounded to the correct number of significant figures).

For soil samples subjected to GPC clean-up procedures, the extract must be concentrated to 0.5 mL, and the sensitivity of the analysis is not compromised by the cleanup procedures. Therefore, the CRQL values in Exhibit C will apply to all samples, regardless of cleanup. However, if a sample extract cannot be concentrated to the protocol-specified volume (see Exhibit C), this fact must be accounted for in reporting the sample quantitation limit.

J - Indicates an estimated value. This flag is used either when estimating a concentration for tentatively identified compounds where a 1:1 response is assumed, or when the mass spectral data indicate the presence of a compound that meets the identification criteria but the result is less than the sample quantitation limit but greater than zero. For example, if the sample quantitation limit of 10 µg/L, but a concentration of 3 µg/L is calculated, report it as 3J. The sample quantitation limit must be adjusted for dilution as discussed for the U flag.

N - Indicates presumptive evidence of a compound. This flag is only used for tentatively identified compounds, where the identification is based on a mass spectral library search. It is applied to all TIC results.

P - This flag is used for a pesticide/Aroclor target analyte when there is greater than 25% difference for detected concentrations between the two GC columns (see Form X). The lower of the two values is reported on Form I and flagged with an "P".

C - This flag applies to pesticide results where the identification has been confirmed by GC/MS. If GC/MS confirmation was attempted but was unsuccessful, do not apply this flag, instead use a laboratory-define flag, discussed below.

B - This flag is used when the analyte is found in the associated blank as well as in the sample. It indicates possible/probable blank contamination and warns the data user to take appropriate

action. This flag must be used for a TIC as well as for a positively identified target compound.

E - This flag identifies compounds whose concentrations exceed the calibration range of the GC/MS instrument for that specific analysis. If one or more compounds have a response greater than full scale, except as noted in Exhibit D, the sample or extract must be diluted and re-analyzed according to the specifications in Exhibit D. All such compounds with a response greater than full scale should have the concentration flagged with an "E" on the Form I for the original analysis. If the dilution of the extract causes any compounds identified in the first analysis to be below the calibration range in the second analysis, then the results of both analyses shall be reported on separate copies of Form I. The Form I for the diluted sample shall have the "DL" suffix appended to the sample number. NOTE: For total xylenes, where three isomers are quantified as two peaks, the calibration range of each peak should be considered separately, e.g., a diluted analysis is not required for total xylenes unless the concentration of either peak separately exceeds 200 µg/L.

D - This flag identifies all compounds identified in an analysis at a secondary dilution factor. If a sample or extract is re-analyzed at a higher dilution factor, as in the "E" flag above, the "DL" suffix is appended to the sample number on the Form I for the diluted sample, and all concentration values reported on that Form I are flagged with the "D" flag. This flag alerts data users that any discrepancies between the concentrations reported may be due to dilution of the sample or extract.

A - This flag indicates that a TIC is a suspected aldol-condensation product.

X - Other specific flags may be required to properly define the results. If used, they must be fully described, and such description attached to the Sample Data Summary Package and the SDG Narrative. Begin by using "X". If more than one flag is required, use "Y" and "Z" as needed. If more than five qualifiers are required for a sample result, use the "X" flag to combine several flags, as needed. For instance, the "X" flag might combine the "A", "B", and "D" flags for some sample. The laboratory-defined flags are limited to the letters "X", "Y", and "Z".

The combination of flags "BU" or "UB" is expressly prohibited. Blank contaminants are flagged "B" only when they are detected in the sample.

C. Surrogate Recovery (Form II and PEST)

C.1. Form II is used to report the recoveries of the surrogate compounds added to each pesticide/Aroclor sample, blank, matrix spike, and matrix spike duplicate.

C.2. Complete the header information and enter EPA Sample Numbers as described in part A. For semivolatile soil samples only, specify the "level" as "LOW" or "MED", as on Form I. Do not mix low and medium level samples on one form. Complete one for each level. For each surrogate, report the percent recovery to the nearest whole percentage point, and to the number of significant figures given by the QC limits at the bottom of the form.

C.3. Flag each surrogate recovery outside the QC limits with an asterisk (*). The asterisk must be placed in the last space in each appropriate column, under the "#" symbol. In the far right-hand column, total the number of surrogate recoveries outside the QC limits for each sample. If no surrogates were outside the limits, enter "O".

C.4. If the surrogates are diluted out in any analysis, enter the calculated recovery, or "O" (zero) if the surrogate is not detected, and flag the surrogate recoveries with a "D" in the column under the "#" symbol. Do not include results flagged "D" in the total number of recoveries for each sample outside the QC limits.

C.5. The pesticide surrogate recoveries must be reported from both GC columns used for the analyses. Therefore, identify each GC column in the header, entering the stationary phase under "GC Column" (previously called GC Column ID), and the internal diameter (ID) of the column in millimeters under "ID". The assignment of columns as "1" and "2", is left to the discretion of the laboratory if the analyses are performed by simultaneous injection into a GC containing two columns. If so analyzed, the assignment of "GC Column 1" and "GC Column 2" must be consistent across all the reporting forms. If the analysis is not performed by simultaneous injection, then the assignment of GC Column number should be based on the chronological order of the two analyses.

C.6. The pesticide surrogate recovery limits are only advisory, but the Contractor must flag those recoveries outside the advisory QC limits or diluted out, nonetheless.

C.7. Number all pages as described in part A.

D. Matrix Spike/Matrix Spike Duplicate Recovery (Form III)

D.1. This form is used to report the results of the analyses of a matrix spike and matrix spike duplicate. The form is matrix-specific for volatiles and semivolatiles.

D.2. Complete the header information as instructed in Part A, including the EPA Sample Number for the matrix spike, without the suffixes MS or MSD.

D.3. All water samples are "LOW". Therefore, there is no MS/MSD for "medium level waters", and none shall be reported.

D.4. In the upper box in Form III, under "SPIKE ADDED", enter the calculated concentration in µg/L or µg/Kg (according to the matrix) that results from adding each spiked compound to the aliquot chosen for the matrix spike (MS). For instance, for base/neutral compounds in medium level soils, if 50 µg of spike are added to 1 g of soil, the resulting concentration is 50,000 µg/Kg. Enter the "SAMPLE CONCENTRATION", in 50,000 similar units, of each spike compound detected in the original sample. If a spike compound was not detected during the analysis of the original sample, enter the sample result as "O" (zero). Under "MS CONCENTRATION", enter the actual concentration of each spike compound detected in the matrix spike aliquot. Calculate the percent recovery of each spike compound in the matrix spike aliquot to the nearest whole percent, according to Exhibit E, and enter under "MS % REC". Flag all percent recoveries outside the QC limits with an asterisk (*). The asterisk must be placed in the last space of the percent recovery column, under the "#" symbol.

D.5. For pesticide/Aroclor matrix spikes and matrix spike duplicates, the concentration used for "MS CONCENTRATION" AND "MSD CONCENTRATION" must be the concentration of the spiked analyte reported on Form I that those analyses. Of the two concentrations calculated for each pesticide/Aroclor target compound, one on each GC column, the lower concentration is reported on Form I, and both concentrations are reported on Form X. The lower concentration is reported on Form III and used in the calculation of spike recovery, even if that concentration yields a recovery value that is outside the advisory QC limits.

D.6. Complete the lower box on Form III in a similar fashion, using the results of the analysis of the matrix spike duplicate (MSD) aliquot. Calculate the relative percent difference (RPD) between the matrix spike recovery and the matrix spike duplicate recovery, and enter this value in the lower box under "% RPD". Report the relative percent difference to the nearest whole percent. Compare the RPDs to the QC limits given on the form, and flag each RPD outside the QC limits with an asterisk (*) in the last space of the "% RPD" column, under the "#" symbol.

D.7. Summarize the values outside the QC limits at the bottom of the page. No further action is required by the laboratory. Performance-based QC limits will be generated and updated from recovery and RPD data.

E. Method Blank Summary (Form IV)

E.1. This form summarizes the samples associated with each method blank analysis. A copy of the appropriate From IV is required for each blank.

E.2. Complete the header information on Form IV as described in Part A. The "EPA Sample No." entered in the box at the top of Form IV shall be the same number entered on the Form I for the blank itself.

E.3. For pesticide/Aroclor blanks, enter the method of extraction as "SEPF" for separator-y funnel, "SONC" for sonication, or "CONT" for continuous liquid-liquid extraction.

E.4. For pesticide/Aroclor blanks, there is no differentiation between medium and low level soil samples, so no "Level" is entered on this form.

E.5. For pesticide/Aroclor method blanks, enter the date of extraction of the blank.

E.6. If the samples associated with pesticide/Aroclor blank are subjected to sulfur cleanup, then the blank must also be subjected to sulfur cleanup. If sulfur cleanup is employed, enter "Y" in the "Sulfur Cleanup" field, else, enter "N". If only some of the samples associated with the method blank are subjected to sulfur cleanup, a separate sulfur cleanup blank is required (see Exhibit D PEST). If a separate sulfur cleanup blank is prepared, complete one version of Form IV associating all the samples with the method blank, and a second version of Form IV listing only those samples associated with the separate sulfur cleanup blank. Note: Subjecting all samples associated with a method blank to sulfur cleanup avoids the need for two forms.

E.7. Pesticide/Aroclor contaminants must meet the identification criteria in Exhibit D PEST, which requires analysis of the blank on two different GC Columns. Therefore, enter the date, time and instrument ID of both analyses of the blank on the pesticide method blank summary. The information on the two analyses is differentiated as Date Analyzed (1), Date Analyzed (2), etc. If the analyses were run simultaneously, the order of reporting is not important, but must be consistent with the information reported on all other pesticide forms. Otherwise, (1) shall be the first analysis, and (2) the second. Identify the GC Column and internal diameter as described previously.

E.8. Enter "Lab File ID" only if GC/MS confirmation was attempted. otherwise, leave blank.

E.9. For all three fractions, as appropriate, summarize the samples associated with a given method blank in the table below the header, entering EPA Sample Number and Lab Sample ID. For volatiles, enter the Lab File ID and time of analysis of each sample. For semivolatile, enter the Lab File ID and Date Analyzed. For pesticides/Aroclors, enter the dates of both analyses as Date Analyzed (1) and Date Analyzed (2), as discussed above.

E.10. Number all pages as described in part A.

F. GC/EC Initial Calibration Data (Form VI PEST-1, PEST-2, PEST-3, PEST-4)

F.1. The initial calibration of pesticides and Aroclors involves the determination of retention times, retention time window, and calibration factors. For single component pesticide target compounds, these data are calculated from the analyses of the Individual Standard Mixtures A and B at three different concentration levels. For the multicomponent target compounds, these data are calculated from a single point calibration.

F.2. For each set of three analyses of Individual Standard Mixture A (low point, mid point, and high point), and set of three analyses of Individual Standard Mixture B, during an initial calibration, complete one copy of Form VI for each GC column used. Thus, each initial calibration will require at least two forms for the Individual Mixture A analyses, and two for Individual Mixture B analyses. However, for each of the forms, half of the compounds will have no entries, as they are not in that mixture.

F.3. Complete the header information as above. Enter the Instrument ID, GC Column, and ID as described previously. Enter the dates of analysis of the first and last of the three standards on each form under "Date(s) Analyzed". Under "Level (x low)", enter the concentration of the low point, mid point, and high point calibration standards as a multiplier of the low point. Therefore, for the low point, enter "1.0". The concentration of the mid point standard is specified in Exhibit D as ten times the low point, therefore, enter "4.0" for "mid". The high point standard must be at least 16 times the low point, but may be higher, if that value lies within the linear range of the instrument, as specified in Exhibit D. Therefore, enter the appropriate multiplier to the high point standard concentration to one decimal place.

F.4. For each standard analyzed, enter the retention time of each applicable analyte in minutes and decimal minutes, under the appropriate concentration level. Calculate the mean retention time of each analyte from the three individual mixtures, and report it under "Mean RT". Calculate the retention time window for each analyte, using these specifications in Exhibit D, and enter the lower limit of the window under RT Window "From", and the upper limit of the window under "To". The retention times of the surrogates are reported for both Individual mixtures, but the windows are only required to be calculated for individual Mixture A.

F.5. For each three analyses of the same Individual Standard Mixture (A or B), the laboratory must also complete the calibration factor data on Form VI PEST-2. In a similar fashion as for the retention time data on Form VI PEST-1, prepare one form for each group of three standards, for each instrument and GC column used. Enter the concentration level of the standards in the same fashion as on Form VI PEST-1.

F.6. Enter the calibration factor for each compound in each of the standards, and calculate a mean calibration and a percent relative standard deviation (%RSD), and enter on the form.

As with surrogate retention times, the calibration factors are only required from Individual Mixture A analyses.

F.7. In order to be used for sample analyses, the %RSD of the initial calibration factors must be less than or equal to 20.0 percent, (25.0 % for alpha-BHC and delta-BHC), except as noted in the following. The %RSD of the calibration factors for the two surrogates must be less than or equal to 30.0 percent. Up to two single component target compounds (but not surrogates) may exceed the 20.0 percent limit for %RSD, (25.0% for alpha-BHC and delta-BHC) but these compounds must have a %RSD of less than or equal to 30.0 percent. These criteria apply to both GC columns.

F.8. For the multicomponent target compounds, the retention times, retention time windows, and calibration factor must be reported in a similar fashion for each single point calibration standard. For each multi-component compound, the laboratory must select at least three peaks from each analyte, according to the specifications in Exhibit D. The retention and calibration factor data apply to each peak. Complete one version of Form VI PEST-3 for each GC column, for each initial calibration that applies to samples in the data package.

F.9. Form VI is used also to report the results of analysis of the Resolution Check Solution that must begin each pesticide/Aroclor initial calibration sequence. The purpose of the Resolution Check Solution is to demonstrate for each initial calibration that the GC columns employed are capable of satisfactorily resolving the most difficult of the target analytes. One copy of Form VI PEST-4 is completed that covers both GC columns.

F.10. Complete the header information as described in Instruction A. Using the same assignment of first and second GC columns made for Form IV, enter the GC Column, ID, Instrument ID, and Data and Time Analyzed. Enter the "EPA Sample No." for the Resolution Check Standard. If simultaneous injections on a single GC are used, the EPA Sample No. may be the same for both Resolution Check Standards. If simultaneous injections were not used, use different suffixes to identify the standards.

F.11. In the boxes on the form, list each analyte, in retention time order, including both surrogate compounds. Thus, the order of analytes in the two boxes on a copy of this form will be different, due to the dissimilarity of the stationary phases of the two GC columns used. Enter the name of each target analyte in the Resolution Check Mixture as it appears on Form I PEST. Spell out the names of the surrogates as they appear on Form VI PEST-2.

F.12. Enter the retention time of each analyte from the analysis under "RT". Calculate the resolution between each pair of analytes according to the formulae in Exhibit D. The resolution is calculated as percentage of the height of the smaller of each pair of adjacent peaks. Enter the resolution between the first and second peaks on the line for the first analyte listed in the box. Enter the resolution between the second and third peaks on the line

for the second analyte, and so on, until the resolutions of all possible pairs of adjacent analytes have been entered. NOTE: Only eight of the nine resolution fields will be filled. In order for these GC columns to be used for pesticide/Aroclor analyses, the resolution of all pairs of peaks listed on this form must be greater than or equal to 60.0%.

G. GC/EC Continuing Calibration (Form VII PEST)

G.1. The calibration verification Summary Form VII is used to report the results of the Performance Evaluation Mixtures (PEM), instrument blanks, and Individual Standard Mixtures A and B analyzed at the beginning and end of a twelve hour sequence. The laboratory must submit this form for each twelve hour sequence analyzed.

G.2. Complete the header information on each Form VII required according to the instructions in part A.

G.3. Enter the initial calibration date(s) analyzed. Give inclusive dates if initial calibration is performed over more than one date.

G.4. On Form VII PEST-1, enter the EPA Sample No., Lab Sample ID, Date Analyzed, and Time Analyzed for the instrument blank that preceded the twelve hour sequence (PIBLK). For the PEM that initiated or terminated the twelve hour sequence (PEM), enter the EPA Sample No., Lab Sample ID, Date Analyzed, and Time Analyzed.

G.5. In the table, report the retention time for each analyte in the PEM as well as the retention time windows. For each analyte in the PEM, enter the amount of the analyte calculated to be in the PEM, in nanograms to three decimal places, under "CALC AMOUNT". Enter the nominal amount of each analyte in the PEM under "NOM AMOUNT". Calculate the relative percent difference between the calculated amount and nominal amount for each analyte according to Exhibit D. Report the values under "RPD". Calculate the percent breakdown for endrin and 4,4'-DDT, and the combined percent breakdown in the PEM according to Exhibit D. Enter the values for the breakdown of endrin and 4,4'-DDT in their respective fields immediately under the table.

G.6. Form VII PEST-2 is used to report the results of the analyses of the instrument blank and the midpoint concentrations of Individual Standard Mixtures A and B that, along with the PEM, bracket each 12-hour period of sample analyses. One copy of Form VII PEST-2 must be completed each time the Individual Standard Mixtures are analyzed, for each GC column used. The form is completed in a fashion similar to From VII, entering the EPA Sample No., Lab Sample ID, Date Analyzed, and Time Analyzed for the instrument blank immediately preceding the Individual Standard Mixtures A and B, and for the standards themselves. The upper table on the form contains the retention time and amount data for Individual Standard Mixture A compounds. The lower table contains the data for Mixture

B. enter the data in these tables in a fashion similar to that for the PEM. Complete copies of Form VII PEST-1 and PEST-2 for each standard reported on Form VIII PEST.

H. Pesticide Analytical Sequence (Form VIII Pest)

H.1. This form is used to report the analytical sequence for pesticide analysis. At least one Form VIII PEST is required for each GC column used for pesticide/Aroclor analyses.

H.2. The laboratory shall complete all the header information as in Part A. Enter dates of analyses for the initial calibration, GC column, ID, and Instrument ID, as on Forms IV, VI, and VII.

H.3. At the top of the table, report the mean retention time for tetrachloro-m-xylene and decachlorobiphenyl calculated from the initial calibration sequence under "TCX" and "DCB", respectively. For every analysis associated with a particular analytical sequence starting with the initial calibration, enter the EPA Sample Number, Lab Sample ID, Date Analyzed, and Time Analyzed. Each sample analyzed as part of the sequence must be reported on Form VIII PEST even if it is not associated with the SDG. The laboratory may use the EPA Sample No. of "ZZZZZ" to distinguish all samples that are not part of the SDG being reported. Report the retention time of the surrogates for each analysis under "TCX RT" and "DCB RT". All sample analyses must be bracketed by acceptable analyses of instrument blanks, a PEM, and Individual Standard Mixtures A and B. Given the fact that the initial calibration may remain valid for some time (see Exhibit D), it is not necessary to report the data from 12-hour periods when no samples in an SDG were run. The laboratory must deliver the Form VIII for the initial calibration sequence, and Forms that include the PEMs and Individual Standard Mixtures that bracket any and all samples in the SDG. While the data for time periods between the initial calibration and samples in the SDG is not a routine deliverable, it must be made available on request during on-site evaluations, etc. Here again, non-EPA samples may be indicated with "ZZZZZ".

H.4. Flag all those values which do not meet the contract requirements by entering an asterisk (*) in the last column, under the "*". If the retention time cannot be calculated due to interfering peaks, leave the RT column blank for that surrogate, enter an asterisk in the last column, and document the problem in the SDG Narrative.

H.5. If more than a single copy of Form VIII PEST is required, enter the same header information on all subsequent pages for that GC Column and Instrument, and number each page as described in Part A.

H.6. Form VIII PEST is required for each for each GC system and for each GC column used to analyze target pesticides/Aroclors.

I. Pesticide Cleanup Summary (Form IX PEST-1, PEST-2)

I.1. This form summarizes the results of the checks performed for both cleanup procedures employed during the preparation of pesticide extracts for analysis. Form IX PEST-1 is used to report the results of the check of the Florisil cartridges used to process all sample extracts, and to associate the lot of cartridges with particular sample results. In this fashion, problems with a lot of cartridges may be tracked across many sample.

I.2. Complete the header information on each Form IX required, according to the instructions in Part A.

I.3. Enter the "Case No." and "SDG No." for the current data package, regardless of the original Case for which the cartridge check was performed. Enter the "Florisil Cartridge Lot Number". Enter under the "Date Analyzed" the date the Florisil cartridge check solution was analyzed.

I.4. Enter "GC Column" and "ID" for the GC columns used to determine the recovery of the analytes in the Florisil cartridge check solution, under "GC Column (1)", and "GC Column (2)", etc., as discussed previously.

I.5. In the upper table, enter the amount of spike added and spike recovered in nanograms for each analyte.

I.6. Calculate to the nearest whole percent, and enter the percent recovery in the "% REC" field. Flag each spike recovery outside the QC limits with an asterisk (*). The asterisk must be placed in the last space in the "% REC" column, under the "#" symbol.

I.7. In the lower table, enter the "EPA Sample No.", the "Lab Sample ID", and "Date Analyzed" for each sample and blank that was cleaned up using this lot of Florisil cartridges.

I.8. Number the Form IX pages as described in Part A.

I.9. Form IX PEST-2 summarizes the results of the calibration of the Gel Permeation Chromatography device (GPC) that must be used to process all soil sample extracts for pesticide/Aroclor analyses. Calibration of the GPC is required at least once every 7 days, and each time the GPC column is repacked.

I.10. Complete all header information as in Part A. Enter an identifier for the GPC Column, and the date of calibration in the appropriate fields. Enter the two "GC Column" and "ID" fields, as discussed above.

I.11. For each of the pesticide matrix spike compounds listed in the box in the upper portion of the form, enter the amount of the spike added to the GPC column in ng, and the amount recovered, also in ng. Calculate the percent recovery of each analyte, and enter these values on the form, to the nearest percent. Compare the recoveries to the QC limits shown on the form, and flag all those values outside the limits with an asterisk (*) in the column under the "#" symbol.

I.12. For each sample in the data package that was subjected to GPC under this calibration, enter the EPA Sample No., Lab Sample ID, and the date of both analyses in the lower portion of the form.

I.13. If more than one copy of Form IX PEST-2 is required, number all pages as described in Instruction A.

J. Pesticide/Aroclor Identification (Form X PEST-1, PEST-2)

J.1. This form summarizes the quantitations of all target pesticides/Aroclors detected in a given sample. It reports the retention times of the compound on both columns on which it was analyzed, as well as the retention time windows of the standard for that compound on both of these columns. In addition, it is used to report the concentration determined from each GC column, and the percent difference between the two quantitative results. Separate copies of Form X are used for single component analytes and multicomponent analytes.

J.2. Copies of Form X are required for each sample, blank, matrix spike, and matrix spike duplicate in which target pesticides or Aroclors are detected. If none are detected in a given sample, no copy of Form X is required for that sample.

J.3. Compete the header information as in Instruction A. Enter the GC Column, and ID for each of the two columns, one as GC Column (1), the other as (2), as described previously. Enter the Instrument ID associated with each GC column directly below.

J.4. For each single component pesticide detected, enter the name of the compound under "ANALYTE" as it appears on Form I. Enter the retention times on each column of the compounds detected in the sample next to the appropriate column designation (1 or 2). Enter the retention time windows on each column from the initial calibration standard. These data must correspond with those on Form VI, and are entered in a similar manner. The lower value is entered under the "FROM" column, the upper value under the "TO" column.

J.5. Enter the concentration calculated from each GC column under the column labeled "CONCENTRATION". The units are the same as those used on From I, µg/L for water sample, and µg/Kg for soil samples. However, do not enter any units on Form X.

J.6. Calculate the percent difference between the concentrations entered, and report it to a tenth of a percent under "%D".

J.7. The lower of the two concentrations is reported on Form I for each pesticide compound. The lower concentration is used because, if present, co-eluting interferences are likely to increase the calculated concentration of any target compound. If the percent difference between the calculated concentrations is greater than 25.0 percent, flag the concentration on From I, as described previously. This will alert the data user to the potential problems in quantitating this analyte.

J.8. If more pesticide compounds are identified in an individual sample than can be reported on one copy of Form X, then complete as may additional copies of Form X as necessary, duplicating all header information, and numbering the pages as described in Instruction A.

J.9. Multicomponent analytes detected in samples are reported on a separate version of Form X. Complete the header information and Instrument and GC Column fields as described above. For multicomponent analytes, it is necessary to report the retention time and concentration of each peak chosen for quantitation in the target analyte, in fashion similar to that for single component pesticides. The concentrations of all peaks quantitated (three are required, up to five may be used) are averaged to determine the mean concentration. Report the lower of the two mean concentrations on Form I. Flag this value as described previously, if the mean concentrations from the two GC columns differ by more than 25.0 percent.

J.10. If more multicomponent compounds are identified in an individual sample than can be reported on one copy of Form X, then complete as many additional copies of Form X as necessary, duplicating all header information, and numbering the pages as described in Instruction A.

Laucks Testing Laboratories, Inc. 4/9/97
Addendum to Laucks SOP LTL-8082, Revision 3

We are proposing the following deviations from OLM03.1 for the CLP analyses of PCBs in Soil Water samples for the NIROP - Fridley, Minnesota Project

Since we will not be analyzing these samples for pesticides, we are proposing that the pesticide portion of the run sequence be eliminated and the following modifications be performed

Sequence and Calibration:

Initial Calibration

Aroclor 1221@ 200 ng/mL
Aroclor 1232@ 100 ng/mL
Aroclor 1242@ 100 ng/mL
Aroclor 1248@ 100 ng/mL
Aroclor 1254@ 100 ng/mL
Aroclor 1016/1260 LOW@ 100 ng/mL
Aroclor 1016/1260 MID@ 500 ng/mL
Aroclor 1016/1260 HIGH@ 1000 ng/mL
•
•
10 samples
•
•
Continuing Calibration
Aroclor 1016/1260@ 100 ng/mL

Standard and Surrogate Information:
Each of the 100 and 200 ng/mL PCB standards would contain the surrogates TCMX and DCB at 20 ng/mL. The Aroclor 1016/1260 MID level standard would contain the surrogates at 10 ng/mL and the Aroclor 1016/1260 HIGH level standard would contain the surrogates at 100 ng/mL

Quality Control:
All water samples and QC would be spiked with 1 mL of a 200 ng/mL TCMX/DCB surrogate solution (or equivalent solutions producing a 200 ng spike amount) and soil samples would be spiked with 2 mL of the surrogate solution.

Matrix Spike/Matrix Spike duplicate samples would be spiked with 1 mL of a 5 ug/mL Aroclor 1016/1260 spike solution instead of with the normal pesticide spiking solution.

Additionally, a laboratory spike control sample, containing Aroclor 1016/1260, will be processed with each batch.

Quantitation:
All sample quantitations will be based upon single point standards as specified in OLM03.1. Dilutions will be performed whenever the calculated concentration exceeds 10X the concentration of the low level PCB standard.

Data Package:
The following forms would not be included:

6D & 6E "PESTICIDE INITIAL CALIBRATION OF SINGLE COMPONENT ANALYTES
6G "PESTICIDE ANALYTE RESOLUTION SUMMARY"
6H "PERFORMANCE EVALUATION MIXTURE (PEM)"
7D & 7E "PESTICIDE CALIBRATION VERIFICATION SUMMARY"
9A & 9B "PESTICIDE FLORISIL CARTRIDGE CHECK AND PESTICIDE GPC CALIBRATION"
10A "PESTICIDE ID SUMMARY FOR SINGLE COMPONENT ANALYTES"

A new form would be included that documents the linearity of the surrogate compounds and Aroclor 1016/1260 mix at three concentration levels. This would be equivalent to a form 6.

A new multicomponent continuing calibration form would be generated. This would be equivalent to a form 7.

The above procedural changes will supersede the Laucks SOP LTL-8082 for the duration of the project.

Reviewed by Monica Carr, Laucks Organic Supervisor	4/9/97 Date
Approved by Harry Romberg, Laucks QA Manager	4-9-97 Date
Approved by Daneen Resnick, Brown & Root	4/11/97 Date
Other Approval	Date

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

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UNCONTROLLED

itle: Determination of Volatile Organic Compounds by Method SW 8260A

ritten by: Monica Carr, Organic Division Manager Date: 4/9/97

pproved by: Harry Romberg, QCO Date: 4-9-97

pproved by: Karen Kotz, Lab Director Date: 4/9/97

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I I - Introduction and Scope

t 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 in addition to low level water samples. A 25 mL sample volume is employed in order to achieve lower detection limits for the low level waters.

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.

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.

: 2. **Method Deviations**

Detailed below are any deviations from the published version of method SW 8260A. All deviations are followed as the standard operation procedure by Laucks Testing Labs.

The suggested use of an alternative calibration curve using regression fit in instances when the recommended %RSD limit of 15 is exceeded for the Calibration Check Compounds (CCCs) is not employed. All calibrations are based on the use of relative response factors.

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. Refer to section VI for detailed method blank criteria.

: 3. **Sample Collection, Sample Storage, Holding Times**

Samples are collected with zero headspace in 40 mL glass containers for waters and 2 ounce glass containers for soils and sealed with Teflon-lined caps. The aqueous samples are preserved to a pH of <2 with HCl and all samples are stored at 4°C. The holding time to analysis is 14 days from date of collection. The pH values for all aqueous samples are measured subsequent to sample analysis and recorded in the instrument logbook.

Part 4. **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.

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 NEESA 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 the same acronym used in the CLP program. This is a standard analyzed 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.

CLP	Contract Laboratory Program. The USEPA program that contracts with laboratories to provide laboratory services. The term has come to mean a much broader set of methods and deliverables. In the context of this SOP, CLP means procedures or operations which are detailed in the CLP contract and which are extended to a broader working definition.
Corr Coef, CC	Correlation coefficient. A measure of the "goodness of fit" of a set of data to a regression model. The closer the value is to 1, the higher the degree of confidence in the correlation.
IBLK	Instrument blank. This term is borrowed from CLP. An instrument blank is PFW containing the method surrogates and is introduced into the instrument to monitor for carry over between sample analyses.
ICV	Initial calibration verification. This term is borrowed from the CLP GC/MS protocol. It is a standard which is analyzed at the beginning of each QC period that is compared to the initial multi-point calibration to determine whether the instrument is still in calibration.
IDL	Instrument detection limit. The lowest concentration of a target analyte that will yield a signal:noise ratio of at least 3x. Used as a starting point for selecting MDL study spiking levels.
MDL	Method detection limit. The lowest concentration in a sample which will yield a positive result that is greater than zero at a known level of confidence. MDLs are empirically determined by Laucks.
MDL standard	Method detection limit standard. A standard prepared so that the concentrations of the target analytes are approximately 4x the empirically determined MDLs. This standard is used to verify that the instrument is capable of detecting the target analytes on an ongoing basis.

PFW	Purge-free water. Laboratory deionized water that is boiled for 15 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.
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 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.

Part 5. Note on Using Spreadsheets for Standard Deviation Calculations

LOTUS computes the population standard deviation when using the @STD() function. This value must be multiplied by $\sqrt{(n/(n-1)}$ 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%. If using Quattro Pro, use the @STDS() function to correctly calculate the sample standard deviation. Excell uses the =STDEV() function to calculate the sample standard deviation.

SECTION II - Equipment List and Standards

Part 1. Chromatographic System

Gas Chromatograph	: Hewlett Packard 5890 I or II employing a low-dead-volume interface from the sample concentrator to the GC injection port.
Carrier Gas	: Helium 99.995% (high purity grade) or better.
Column	: 30m or 75m x 0.53mm x 3.0μ film bonded phase fused silica capillary column (J&W DB-624 or equivalent).
Purge and Trap	: OI Analytical 4460A sample concentrator or equivalent.
Autosampler	: Dynatech Precision Sampling PTA-30 W/S or equivalent.
GC/MS Interface	: Hewlett Packard 59913A jet separator assembly or equivalent.

Mass Spectrometer	:	Hewlett Packard 5970B with 59824A scanning interface or equivalent.
Data System	:	Teknivent
Trap	:	OI Corporation, Supelco or equivalent. Examples are listed below. 1. 1cm 3% SP-2100/Chromosorb W AW, 7.7cm Tenax TA, 7.7cm silica gel 15, 7.7cm activated charcoal. Supelco Pt No.:2-1139. 2. VOCARB 3000. 10cm Carbopack C, 6cm Carboxen 1000, 1cm Carboxen 1001. Supelco Pt No.: 2-1131. 3. VOCARB 4000. 8.5cm Carbopack C, 10cm Carbopack B, 6cm Carboxen 1000, 1cm Carboxen 1001. Supelco Pt. No.: 2-1143.
Miscellaneous	:	Assorted gas-tight calibrated syringes, pipets, 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.

Part 2. Standards

2.1 Overview of Volatile Standards Preparation

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.

Two standard logbooks are employed to document all volatile standards. One standard logbook is maintained for stock solutions prepared from neat materials; the other standard logbook is maintained for working solutions which include solutions procured from commercial sources, as well as those solutions made inhouse. All standards are assigned a unique identifier to enable cross-referencing of each individual standard back to the supplier's lot number.

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.

Prior to initial use, all analyte concentrations are verified by analyzing the new solution against a current 5-point calibration. Analyte values within 80-120 percent of their nominal value are considered acceptable.

All standard solutions are stored in the VOA freezer at -10°C to -20°C. The stock and intermediate solutions are stored in flame-sealed amber ampules. The current working solutions are stored in Mininert vials, with the exception of the combined internal and surrogate standard solutions which, once opened, are transferred directly to the autosampler syringe. Prior to daily and initial calibration standard preparation, the standard solutions are removed from the freezer and allowed to warm.

Refer to Appendix I for a tabular listing of all standard solutions and their concentrations.

2.2 Preparation of Internal Standards and Surrogate Standards

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. This solution is stored in flame-sealed amber ampules for a time period not to exceed one year.

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. This solution is stored in flame-sealed amber ampules for a time period not to exceed six months. A 1 µL aliquot of this solution is injected via the autosampler into 5 mL (or 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

2.3 Preparation of Matrix Spike Solution

A certified matrix spiking solution which contains 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 working concentration of 25 µg/mL. This solution is stored in flame-sealed amber ampules for a period of six months, or less if loss of analyte is indicated.

Matrix Spike Solution	*Source	*Ref. number
1,1-Dichloroethene	Restek	30005
Benzene		
Trichloroethene		
Toluene		
Chlorobenzene		

* Or equivalent

The working solution is transferred from the flame-sealed ampule to a Mininert vial when put into use. A 40 µL (8 µL for low level waters) aliquot of this solution is spiked into 50 mL of sample, resulting in a spiked concentration of 20µg/L (or 4 µg/L for low level waters) for each analyte.

Working Concentration	Aliquot	Final Volume	Concentration
		Soil, 25 mL Water:	
25 µg/mL	40 µL	50 mL PFW	20 µg/L
		25 mL Water:	
25 µg/mL	8 µL	50 mL PFW	4 µg/L

2.4 Preparation of p-Bromofluorobenzene (BFB) Solution

The preparation of BFB is similar to that of the internal standards and surrogate standards. Refer to section 2.2. for details.

A working solution of 25 ng/µL is prepared and stored in flame-sealed amber ampules.

2.5 Preparation of Initial Calibration Standards

Certified sets of working 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. They are then diluted to appropriate working concentrations through the above described methods.

These solutions are then diluted in methanol to generate working standards at concentrations of 200 µg/mL. This solution is stored in flame-sealed amber ampules for a period of time not to exceed six months.

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.

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 standards are prepared by taking aliquots of the working 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 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	*Ref. 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
Vinyl acetate	Supelco	4-0327

* Or equivalent

WATER (25 mL)

Working Solution	Aliquot	Final Volume	Concentration
200 µg/mL	1 µL	200 mL	1 µg/L
200 µg/mL	2.5 µL	100 mL	5 µg/L
200 µg/mL	5.0 µL	100 mL	10 µg/L
200 µg/mL	6.2 µL	50 mL	25 µg/L
200 µg/mL	12.5 µL	50 mL	50 µg/L

SOIL

Working Solution	Aliquot	Final Volume	Concentration
200 µg/mL	1.5 µL	100 mL	3 µg/L
200 µg/mL	2.5 µL	50 mL	10 µg/L
200 µg/mL	6.25 µL	25 mL	50 µg/L
200 µg/mL	5 µL	10 mL	100 µg/L
200 µg/mL	10 µL	10 mL	200 µg/L

Water (5 mL)

Working Solution	Aliquot	Final Volume	Concentration
200 µg/mL	1.5 µL	100 mL	3 µg/L
200 µg/mL	2.5 µL	50 mL	10 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

Preparation of a Daily Calibration Standard

The concentration of the continuing calibration standard at a level of 10 µg/L for low level waters and 50 µg/L for the soils 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.

SECTION III - Safety precautions

Part 1. **Routine Safety Precautions**

All standards and samples 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.

Flammable solvents such as methanol are stored in the appropriate solvent locker located outside the volatile laboratory.

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.

Part 2. **Waste disposal**

Waste solvents and expired standards are disposed of in the appropriate waste solvent container located in the prep area under the hood.

Solid sample matrices are disposed of in appropriate drums labeled for this specific purpose where they are routinely picked up by a certified agency for final disposal.

SECTION IV - Operation procedures

Part 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.

Part 2. 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 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.

Part 3. Method Validation

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.

Part 4. **Instrument Tuning**

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.

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%		

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.

4.2 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).

The ion abundances and ratios are checked against the criteria detailed below. 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. Persuant to rev. 1, September 1994, alternative tuning criteria may be used (e.g., CLP, 524.2).

Mass		Abundance %
50		15 - 40% of mass 95
75		30 - 60% of mass 95
95		base peak, 100% relative abundance
96	--	5 - 9% of mass 95
173		< 2% of mass 174
174		> 50% of mass 95
175		5 - 9% of mass 174
176		95 - 101% of mass 174
177		5 - 9% of mass 176

Part 5. **Initial Multi-Point Calibration**

Analyze standard solutions using a minimum of 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. Criteria for evaluating these standards are detailed in Section VI.

Part 6. **Continuing Calibration Verification**

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 VI.

Part 7. **Instrument Blank**

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.

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.

8. **Method Blank**

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 VI for method blank acceptance criteria.

Part 9. **Sample Analysis**

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 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.

9.1 Analysis sequence

IBLK (optional)
BFB
CCV
Method blank
Samples
QC

9.1.1 Sample Preparation

The samples are removed from the VOA refrigerator and are allowed to warm to room temperature. Aqueous samples which are received in 40 mL vials are placed onto the autosampler carousel. Soil samples are first gently mixed (no supernatant liquids are removed) and a 5 gram aliquot of soil is weighed out and transferred to the autosampler vials. The autosampler transfers a 5 mL or 25 mL aliquot of sample (or 5 mL of PFW for the soils) into the autosampler syringe, where it injects 1 μL of the internal standard and surrogate standard solution.

The pH of all aqueous 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 recorded in the instrument logbook.

If the concentration of any target analyte exceeds the initial calibration range, the sample must be diluted and reanalyzed.

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.

The soil sample is gently mixed (no supernatant liquid is removed) and a 4 gram aliquot (weighed out to the nearest 0.1 g) is transferred to a tared 15 mL vial. 9.0 mL of methanol and 1.0 mL of system monitoring compound spiking solution (at a concentration of 25 µg/mL) 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 5 mL aliquot of sample into the autosampler syringe, where it injects 1 µL of the internal standard solution.

9.2 Qualitative Identification

Two criteria must be satisfied to verify the identification of target compounds:

1) elution of the sample component within 0.06 RRT of the standard component and,

2) appropriate correspondence between the sample mass spectrum and the standard mass spectrum.

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.

The relative intensities of ions must be within ± 30% between the standard and sample spectrum.

Ions greater than 10% in the sample spectrum but not

present in the standard spectrum is often considered.

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 using the "N" flag. Compounds meeting the identification criteria must be reported with their spectra.

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.

Relative intensities of ions in the reference spectrum > 10% of the most abundant ion should be present in the sample spectrum.

Molecular ions in the reference spectrum should be present in the sample spectrum.

The relative intensities for the major ions should agree within ± 20%.

Ions greater than 10% in the sample spectrum but not present in the standard spectrum should be reviewed for possible background contamination or coelution.

9.3 Compound Quantification

Target compound concentrations are calculated using the following equations:

9.3.1 Aqueous samples

$$\text{Conc } (\mu g/L) = \frac{Ax * Is * Df}{Ais * ARF * Vo}$$

where

Ax = Area of the characteristic ion (EICP) of the analyte.
Ais = Area of the characteristic ion (EICP) of the internal standard .
Is = Amount of the internal standard (ng).
ARF = Average response factor from the ambient temperature purge of the initial calibration.
Vo = Volume of water purged (mL).
Df = Dilution factor. Assume 1.0 for no dilution.

9.3.2 Low soil (on a dry-weight basis)

$$\text{Conc } (\mu g/kg) = \frac{Ax * Is}{Ais * ARF * Ws * D}$$

where

Ax = Area of the characteristic ion (EICP) of the analyte.
Ais = Area of the characteristic ion (EICP) of the internal standard .
Is = Amount of the internal standard (ng).

ARF = Average response factor from the heated purge of the initial calibration.
Ws = Weight of sample added to purge vessel (g)
D = $\frac{100 - \% \text{ moisture}}{100}$

9.3.3 Medium soil (on a dry weight basis)

$$\text{Conc } (\mu g/kg) = \frac{Ax * Is * Vt * 1000 * Df}{Ais * ARF * Va * Ws * D}$$

where

Ax = Area of the characteristic ion (EICP) of the analyte.
Ais = Area of the characteristic ion (EICP) of the internal standard .
Is = Amount of the internal standard (ng).
ARF = Average response factor from the ambient temperature purge of the calibration standard.
Vt = Total volume of the methanol extract in milliliters (10 mL)
Va = Volume of the aliquot of the methanol extract in microliters (μL) added to the reagent water for purging
Ws = Weight of sample added to purge vessel (g)
D = $\frac{100 - \% \text{ moisture}}{100}$
Df = Dilution factor. Assume 1.0 for no dilution.

SECTION V - Reports

Part 1. **Data Packet Organization**

See Appendix II for a check list detailing data packet organization.

Part 2. **Quality Control Reports**

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 and an MS/MSD report.

Part 3. **Control Limits**

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 bound in Laucks' Control Limits Catalog.

Part 4. **Sample Result Reports**

4.1 Data Qualifying Flags

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

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

SECTION VI - Quality Control

Part 1. Initial Calibration

1.1 Criteria

Initial calibration data are evaluated using %RSD and the relative response factors.

RFs are calculated using the equation

$$RF = \frac{Ax}{Ais} * \frac{Cis}{Cs}$$

Ax = Response of target analyte
Ais = Response of internal standard
Cx = Amount of target analyte (ng/mL).
Cis = Amount of internal standard (ng/mL).

%RSDs are calculated using the equation

$$\%RSD = \frac{SD}{RFx} * 100\%$$

Five compounds, the System Performance Check Compounds (SPCCs) are checked to determine that the minimum average relative response factors are met as tabulated below. In addition, the %RSD for the Calibration Check Compounds (CCCs) must be less than 30%.

SPCC Compounds	Min. RF Limit
Chloromethane	0.100
1,1-Dichloroethane	0.100
Bromoform	>0.100
1,1,2,2-Tetrachloroethane	0.300
Chlorobenzene	0.300

CCC Compounds	%RSD Limit
Vinyl chloride	< 30%
1,1-Dichloroethene	< 30%
Chloroform	< 30%
1,2-Dichloropropane	< 30%
Toluene	< 30%
Ethylbenzene	< 30%

For Navy samples, additional criteria of a maximum %RSD of 40% and a minimum RRF of 0.005 for all other reported analytes has been implemented.

1.2 Corrective action

If the criteria are not met, additional standards may be analyzed or appropriate instrument maintenance and analysis of new standards should be performed.

1.3 Documentation

Copies of the initial calibration standards and the calculated RRFs and %RSDs are stored with the raw data.

Part 2. Continuing Calibration Verification

2.1 Criteria

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:

$$\%D = \frac{(RFi - RFc) * 100}{RFi}$$

where RFi = Average RF from Initial Calibration
RFc = RF from CCV standard

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%

For Navy samples, additional criteria of a maximum %D of 40% and a minimum RF of 0.005 for all other reported analytes has been implemented.

2.2 Corrective action

Check calculations, check standard solutions or perform instrument maintenance. Reanalyze the standard.

2.3 Documentation

Copies of the continuing calibration standards and the calculated RFs and %Ds are stored with the raw data.

Part 3. **Instrument Blank**

3.1 Criteria

The criteria used are the same as those used for the method blank control limits.

3.2 Corrective action

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.

3.3 Documentation

Copies of instrument blank analyses are stored with the sample analyses raw data.

Part 4. **Method Blanks**

4.1 Criteria

A method blank is used to verify contamination free reagents and apparatus. A method blank is analyzed following every CCV and prior to sample analysis. Method blank control limits are ≤ reporting limit for

analytes with the exception of the common laboratory solvents such as methylene chloride and acetone. The control limits for these analytes are ≤ 5 x reporting limit.

4.2 Corrective action

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.

Out-of-control surrogate recoveries in the method blank require reanalysis of the method blank.

4.3 Documentation

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.

Part 5. **Blank Spikes**

5.1 Criteria

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.

5.2 Corrective action

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.

5.3 Documentation

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.

Part 6. **Matrix Spike**

6.1 Criteria

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. 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:

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

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

6.2 Corrective action

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.

6.3 Documentation

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.

Part 7. **Matrix Spike Duplicate**

7.1 Criteria

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:

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

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

7.2 Corrective action

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.

7.3 Documentation

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.

Part 8. **Surrogate Recovery**

8.1 Criteria

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:

$$Recovery = \frac{Sm * 100}{Sa}$$

where Sm = concentration of surrogate measured in sample
Sa = concentration of surrogate added

8.2 Corrective Action

Check calculations for possible error. Reanalysis is required for all surrogate recoveries which exceed the established control limits.

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.

8.3 Documentation

The surrogate recoveries for all samples, blanks, and QC analyses are reported electronically via the laboratory's LIMS database.

SECTION VII - References

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), U.S. EPA Office of Solid Waste and Emergency Response, - Method 8260A 3rd ed. 2nd rev. -
Volatile Organic Compounds By Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique, Revision 0, July 1992.

U.S. EPA Contract Laboratory Program, Statement of Work for Organic Analysis, Multi-Media, Multi-Concentration Document Number OLM01.0, March 1990

APPENDIX I

Initial Calibration Concentrations For Low Level Waters (µg/L)

lowest---> ICV/CCV ---> highest

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

Appendix I (continued)

lowest---> ICV/CCV ---> highest

Compound	STD1	STD2	STD3	STD4	STD5
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
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

Appendix I (continued)

lowest---> ICV/CCV ---> highest

Compound	STD1	STD2	STD3	STD4	STD5
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
Iodomethane	1	5	10	25	50
trans-1,4-Dichloro-2-butene	1	5	10	25	50
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
Vinyl acetate	3	10	50	100	200
2,2-Dichloropropane	3	10	50	100	200
cis-1,2-Dichloroethene	3	10	50	100	200
2-Butanone	10	20	50	100	200
Bromochloromethane	3	10	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
1,1-Dichloropropene	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
Dibromomethane	3	10	50	100	200
Bromodichloromethane	3	10	50	100	200

Appendix I (continued)

lowest---> ICV/CCV ---> highest

Compound	STD1	STD2	STD3	STD4	STD5
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
1,3-Dichloropropane	3	10	50	100	200
2-Hexanone	10	20	50	100	200
Chlorodibromomethane	3	10	50	100	200
1,2-Dibromoethane	3	10	50	100	200
Chlorobenzene	3	10	50	100	200
1,1,1,2-Tetrachloroethane	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

APPENDIX II

Data Packet Check List

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

Title: Determination of Vinyl Chloride by Selected Ion Monitoring (SIM)

Part 1. 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.

M/Z	Dwell Time (milliseconds)
61	50
62	400
63	50
64	50

M/Z	Dwell Time (milliseconds)
49	50
51	50
128	100
129	50

M/Z	Dwell Time (milliseconds)
65	50
67	50
102	50
104	50

:t 2. **Standards**

Internal Standard	Surrogate
Bromochloromethane	1,2-Dichloroethane-d4
+	

APPENDIX IV

Additional Appendix IX Compounds And Their Initial Concentrations (µg/L)

lowest---> ICV/CCV ---> highest

Compound	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
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

APPENDIX V

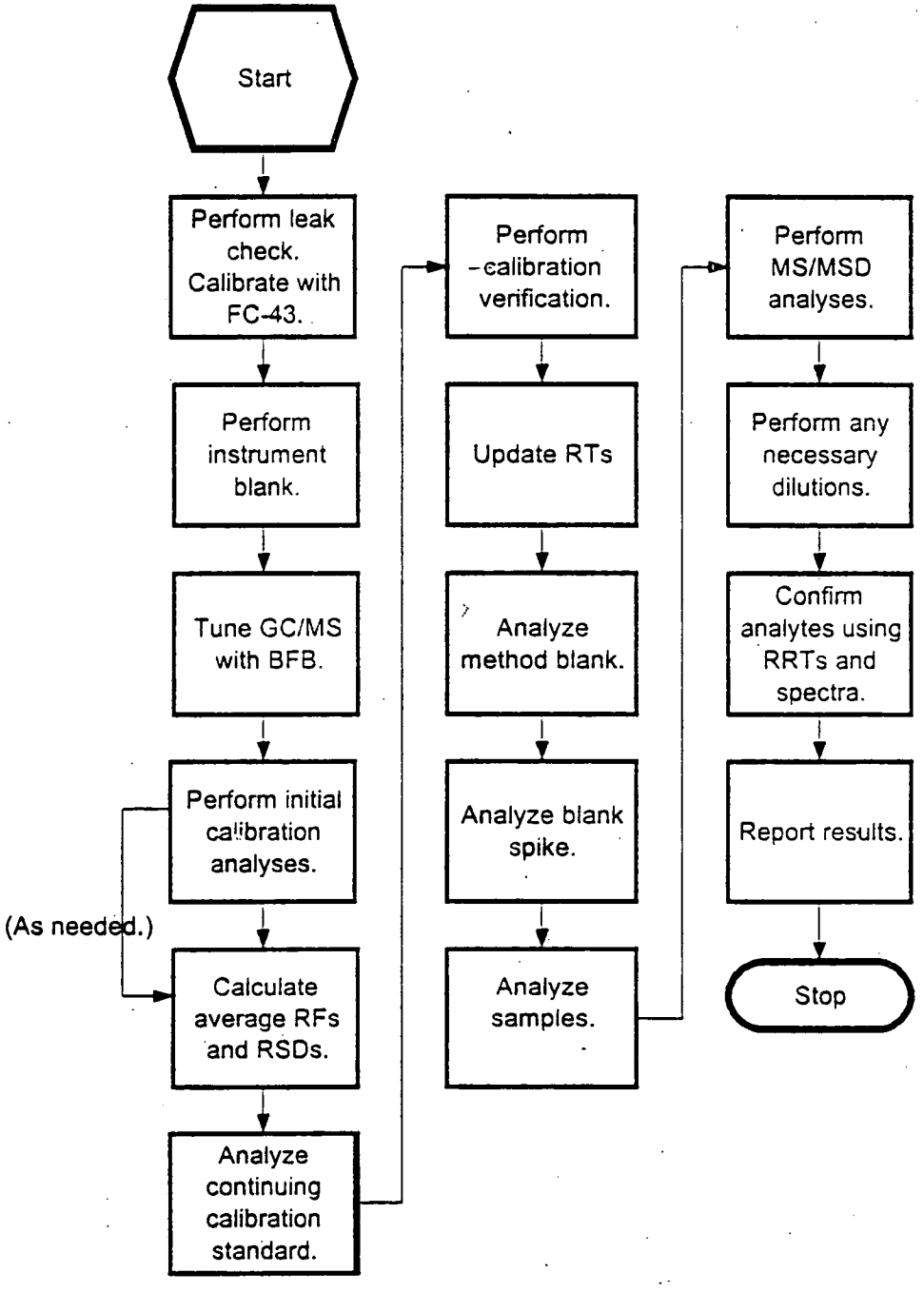
Method 8260 Requirements and Corrective Actions

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 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.	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 MDL. %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	5 levels 1, 5, 10, 20, 50 ug/L. (Ketones 5,10,20,50,100 ug/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 %RSD for all other analytes: 40 percent. Minimum RRF for all other analytes: 0.005.	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.	10 ug/L standard, %D for CCC < 20%, RRF for SPCC > 0.30.	Every 12 hours.	Reanalyze for analytes that exceed the control limits.	Copies of raw data and Form VII.

Internal Standards	1,4-difluorobenzene, chlorobenzene-d5, 1,4-dichlorobenzene-d4 or pentafluorobenzene 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 documented in instrument logbook.
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 flagged in the instrument logbook and 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.	Spike recoveries and RPDs must meet control limits established by laboratory annually. See QC manual 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; ± 0.06 RRT units of standard RRT.	For identifying all target analytes in each sample.		Copies of all mass spectra stored with data.
Sample Preservation and Storage	pH ≤ 2 with HCL or H2SO4 (aqueous). Sodium thiosulfate if residual chlorine (aqueous). Store at 4 ° C.	pH ≤ 2 with HCL. Store at 4° C.	All aqueous samples.	If the aqueous samples are not acid-preserved, this is narrated in final report.	pH of all samples recorded in logbook.

APPENDIX V

METHOD 8260 FLOW CHART



Refer to correctve action chart for out of control events.

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP No. **LTL-9005**

Previous SOP No. **LX-0036**

Title: **Alkalinity (Electrometric) Using EPA Method 310.1**

Rev: 1

UNCONTROLLED

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.

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #: ~~LX-0036~~ 12/26/95
LTL-9005

Title: **Alkalinity (Electrometric) Using EPA Method 310.1**

Revision history:

Number	Date
1.0	06/28/94

Written by: Harry Romberg — Date: 8-2-94
Harry Romberg, Quality Control Officer

Reviewed by: Bill Lundberg — Date: 8/1/94
Bill Lundberg, Inorganic Chemistry Division Manager

Reviewed by: Mike Nelson — Date: 6/30/94
Mike Nelson, Technical Director

Approved by: John M. Buerger — Date: 6/30/94
John Buerger, Laboratory Director

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UNCONTROLLED

1. Introduction and Scope

1.1. Method Description

1.1.1. Scope and Application

1.1.1.1. This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes. It is suitable for all concentration ranges of alkalinity; however, appropriate aliquots should be used to avoid a titration volume greater than 50 ml. Automated titrimetric analysis is equivalent, although it is not available at Laucks at the time of this writing.

1.1.1.2. This method is a potentiometric titration only. It does not address a colorimetric endpoint titration. Additionally, it only addresses total alkalinity, not distinguishing between carbonate, bicarbonate, or hydroxide forms. For methodology which uses a colorimetric endpoint or where a distinction must be made between types of alkalinity, Standard Methods, 18th or later edition, method 2320B should be consulted (Laucks Method LX-0037).

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

1.2. Interferences

1.2.1. Substances, such as salts of weak organic and inorganic acids present in large amounts, may cause interference in the electrometric pH measurements.

1.2.2. For samples having high concentrations of mineral acids, such as mine wastes and associated receiving waters, titrate to an electrometric endpoint of pH 3.9, using the procedure in: Annual Book of ASTM Standards, Part 11.01, "Water", D-1067-88, Method C (page 186 in the 1991 standards book but may appear on other pages in subsequent literature)

1.2.3. Oil and grease, by coating the pH electrode, may also interfere, causing sluggish response if using the electrometric method.

1.3. Sample Collection, Sample Storage, Holding Times

1.3.1. The sample must be refrigerated at 4°C and analyzed as soon as practical, in no case to exceed 14 days from the date of **collection**. The sample bottle should contain minimal head

space and not be opened before analysis. Care should be taken to not aerate the sample, such as by hard shaking of a partially filled bottle, prior to analysis.

1.4. Definition of Terms

1.4.1. This section defines terms and acronyms as they are used in this SOP. Other terms, such as MS/MSD or method blank, are not defined here since it is assumed that the user of this SOP already understands their more general meaning.

1.4.2. 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.

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

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

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

1.4.6. Analytical Sequence - A set of samples and standard solutions analyzed in a chronologically continuous group. See also QC period.

2. Equipment And Reagent Lists

2.1. Equipment:

2.1.1. pH meter or electrically operated titrator that uses a glass electrode and can be read to 0.05 pH units. Standardize and calibrate according to manufacturer's instructions. If automatic temperature compensation is not provided, make titration at 25°C ± 2C°.

2.1.2. Use an appropriate sized vessel to keep the air space above the solution at a minimum.

2.1.3. Magnetic stirrer, pipets, flasks and other standard laboratory equipment.

2.1.4. Burets, Pyrex or equivalent 25 ml.

2.1.5. Graduated cylinders, pipets, Erlenmeyer flasks and other required glassware

2.1.6. Magnetic stirrer and stir bar(s)

2.2. Reagents:

2.2.1. Sodium carbonate solution, approximately 0.05 N: Place 2.5 ± 0.2 g (to nearest mg) primary standard grade Na_2CO_3 (dried at 250°C for 4 hours and cooled in desiccator into a 1 liter volumetric flask and dilute to the mark. The method dictates that this solution not be kept longer than 1 week. This should be adhered to for standardization purposes, but the solution may be kept longer if being used for spiking only, as long as spike control limits are not being exceeded.

2.2.2. 0.02 N Standard Acid - Dilute 5 mls concentrated hydrochloric acid (HCl) to 3 liters with deionized water (DIW). Mix well.

2.2.3. Standardize and adjust normality to .020 ± .001 by potentiometric titration of 15.0 ml 0.05 N Na_2CO_3 solution with about 60 ml distilled water by titrating potentiometrically to pH of about 5. Lift electrode and rinse into beaker. Boil solution gently for 3-5 minutes under a watch glass cover. Cool to room temperature. Rinse cover glass into beaker. Continue titration to the pH inflection point. Calculate normality using:

$$\frac{A \times B}{53. \times C}$$

where:

A = grams Na_2CO_3 weighed into 1 liter
B = ml Na_2CO_3 solution
C = ml acid used to inflection point

2.2.4. Alternatively, (this is a method deviation) standardize by potentiometric titration to a pH of 4.5 against .1000 N sodium hydroxide (NaOH) and adjust to a normality of .020 ± .001. The standardized .1000 N NaOH must be from the buret in the "protein" area as it is traceable to primary standard grade Potassium Biphthalate (KHP).

3. Safety precautions and Waste Disposal

3.1. Safety Precautions

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

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

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

3.2. Waste Disposal

3.2.1. No significant hazardous waste is generated from this procedure. Waste solutions may be disposed of in the laboratory sink, washing with tap water.

4. Calibration and Quality Control

4.1. Method Blanks

4.1.1. Criteria:

4.1.1.1. Method blanks are used to verify contamination free reagents and apparatus. They are prepared with every set of samples prepared at the same time or at least one blank every 20 samples which ever is more frequent. Any analyte response above the detection limit is reported. Method blank control limits for this analysis are 4 mg/L. or twice the detection limit.

4.1.2. Corrective Action:

4.1.2.1. For this analysis, the blank is the first sample analyzed. Therefore, if it exceeds the control limit, no samples should be analyzed until the contamination problem is corrected. In most cases, this will probably only mean re-analyzing another blank.

4.1.2.2. If for some reason samples are analyzed with a blank which exceeds the limit, corrective action may necessitate re-preparation and re-analysis of the sample set. For example, if alkalinity above the limit were found in the blank but not in any of the associated samples, the sample group may not require re-analysis. In addition, if sample levels exceed 10 times the blank, the level of contamination may be considered insignificant. In any case, if re-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 all cases where blank contamination exceeds the control limit a narrative comment must be made which documents the corrective actions taken.

4.2. Standard Reference Material or Laboratory Control Sample

4.2.1. For this analysis, the SRM is also often called an Initial Calibration Verification (ICV). Immediately after the initial blank, analyze a standard from a source other than that from which the calibration material was obtained, preferably an EPA traceable reference material. The reference material must be processed in the same manner as the samples from that run will be, that is either potentiometrically or colorimetrically. The SRM from one method of measurement (potentiometric or colorimetric) will not be used for the other method. One SRM should be processed with every 20 samples or once per analytical run, whichever is greater.

4.2.1. Criteria

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

4.2.2. Corrective action

4.2.2.1. If the ICV (SRM) criteria are not met, no samples can be analyzed. Clean and recalibrate the pH meter and re-check the ICV. If the criteria still cannot be met, the system must be recalibrated, meaning that the normality of the titrant may need to be re-checked. If this is in control, it may be that the SRM solution has deteriorated and another SRM should

be prepared and checked. If the solution is still not in-control, the Division Manager and/or QC Officer should be consulted.

4.3. Sample Duplicate

4.3.1. Criteria

4.3.1.1. At least one duplicate sample per 10 samples is required for this analysis (10% frequency). No matrix spiking is currently required for this analysis. However, if a matrix spike is performed, this frequency may be reduced to one each MS and duplicate per 20 samples.

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

where:

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

4.3.1.2. The RPD control limits for this analysis are detailed in the current Control Limits Catalog and in the Quality Control Database (QC_DB) and will change from time to time. One of these sources should be consulted for the most current control limits.

4.3.2. Corrective action

4.3.2.1. The RPD for this analysis should rarely if ever vary significantly. If it does, it may be an indication of sample inhomogeniety or an error in analytical technique. The source of variation should be determined and, if possible, eliminated. If the source of variation is not controllable, a narrative comment in the data report may be added by the QC Officer or as overseen by the Division Manager. A controllable cause, such as instrument repair or additional training in the analytical technique, should be undertaken if this is thought to be the cause. In either instance, the QC Officer should be consulted and a course of action determined. As a minimum, another aliquot of the QC sample will likely be analyzed and additional actions taken from that point.

5. Operation procedures

5.1. Sample Analysis

5.1.1. Analysis sequence

5.1.1.1. Method Blank
5.1.1.2. SRM (also called an LCS or an ICV for this analysis)
5.1.1.3. up to 20 samples plus QC
5.1.1.4. repeat above sequence

5.2. Instrumental Conditions

5.2.1. The temperature correction probe should be in place and functioning.

5.2.2. The pH meter must first be calibrated with pH 7 and 4 buffer solutions.

5.3. Analytical Operation

5.3.1. Sample size:

5.3.1.1. Use a sufficiently large volume of sample to obtain good precision while keeping volume of titrant low enough to permit a sharp end point. In general, use a 50 ml sample volume except when an alkalinity of <20 mg/l is expected. In this case, a volume of 100-200 mls should be used. A titrant volume of >50 mls should be avoided by reducing the sample volume. The sample should not be diluted or modified in any way. If it is necessary to reduce the sample volume to the extent that it cannot be analyzed using 0.02 N acid and <50 ml titrant volume, it is more appropriate to maintain a larger sample size and use a stronger titrant in order to obtain reliable results.

5.3.2. Potentiometric titration of samples >20 mg/L alkalinity

5.3.2.1. Place sample in flask using the smallest graduated cylinder which will accommodate the size of sample being measured. If less than 25 mls of sample is being dispensed, a graduated glass (Mohr) pipet should be used. The sample should be dispensed with a minimum of aeration so as not to affect the titration due to absorption of atmospheric CO_2. See additional discussion of sample size above.

5.3.2.2. Measure pH of sample

5.3.2.3. Add standard acid, being careful to stir thoroughly but gently to allow pH to equilibrate.

5.3.2.4. Titrate to pH 4.5. Record volume of titrant.

5.3.2.5. If this volume is <1.0 ml, additional sample should be titrated by adding and continuing the titration. If the alkalinity is <20 mg/l, additional sample should be added and the low-level method below should be used.

5.3.3. Potentiometric titration of low alkalinity (<20 mg/L):

5.3.3.1. For alkalinity of <20 mg/1 titrate 100-200 ml as above.

5.3.3.2. Stop titration at pH in range of 4.3-4.7, record volume and exact pH.

5.3.3.3. Very carefully add titrant to lower pH exactly 0.3 pH units and record volume.

5.4. Quantification

5.4.1. Total alkalinity is calculated using the following equations:

5.4.2. Potentiometric titration to pH 4.5

$$\text{Alkalinity, mg/1 } CaCO_3 = \frac{A \times N \times 50{,}000}{\text{ml of sample}}$$

where:
A = ml standard acid
N = normality standard acid

5.4.3. Potentiometric titration of low alkalinity:

$$\text{Total alkalinity, mg/1 } CaCO_3 = \frac{(2B - C) \times N \times 50{,}000}{\text{ml of sample}}$$

where:
B = ml titrant to first recorded pH
C = total ml titrant to reach pH 0.3 units lower
N = normality of acid

6. Reports

6.1. Data Packet Organization

6.1.1. The data package for this analysis consists of the data sheet and a quality control database (QC_DB) report form.

6.2. Quality Control Reports

6.2.1. All results for quality control tests are entered into the lab data base using the QC_DB program. Printouts of all data entered need not be included in the package. However, all must be referenced on the report form. This includes the blank, SRM (ICV), and duplicate results and any other QC which might have been analyzed by special request.

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.

6.3.2. Control Chart(s)

6.3.2.1. The recovery values for the LCS/SRM are plotted on a control chart.

Appendix I

QC Summary Table

Laucks Testing Laboratories

Method EPA 310.1 QA Requirements and Corrective Actions

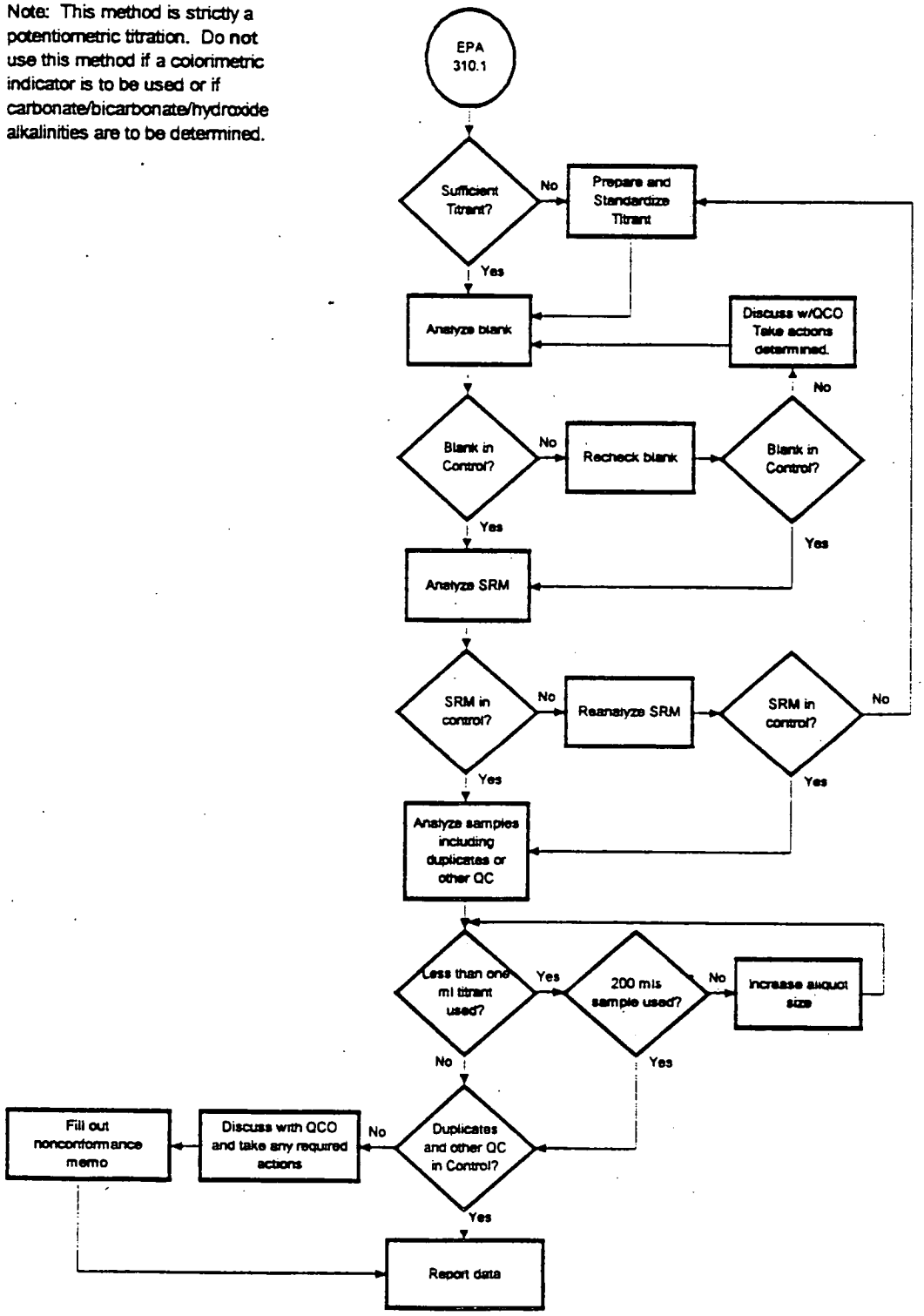
QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Initial Calibration	N/A	Buffer pH meter between pH 4 &7 before analyses	Once before analyzing any samples or QC	None	None
Initial Calibration Verification	Not Required	See SRM			
Continuing Calibration Verification	Not Required	See SRM			
Method Blank	Not Required	<2X the detection limit	5% frequency (1 in 20 samples)	Do not analyze samples until problem is corrected	QC_DB report form with appropriate commentary
Matrix Spike Recovery	Not Required	Not Required			
MS/MSD RPD	Not Required	Not Required			
Duplicate % Difference	Not Required	10% for values >5X the detection limit	10% (1 in 10) (5% if MS is being analyzed)	Correct problem Consult QC Officer	QC_DB report form with appropriate commentary
Blank Spike Recovery	Not Required	Not Required			
Standard Reference Material (SRM) Recovery	Not Required	Within vendor supplied limits or 90%-110% recovery	5% frequency (1 in 20 samples)	Do not analyze samples until problem corrected	QC_DB report form with appropriate commentary

Appendix II

Flow Chart

Alkalinity
EPA 310.1

Note: This method is strictly a potentiometric titration. Do not use this method if a colorimetric indicator is to be used or if carbonate/bicarbonate/hydroxide alkalinities are to be determined.



LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-9009

Title: **Total Hardness by EPA 130.2**

Revision history:

Number	Date
0	03/08/97

UNCONTROLLED

Written by: Bill Lundberg Date: 3/10/97
Bill Lundberg, Conventionals Supervisor

Approved by: Harry Romberg Date: 3-10-97
Harry Romberg, QA Officer

Approved by: Karen J. Kotz Date: 3/10/97
Karen Kotz, Laboratory Director

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METHOD _130.2__ QA REQUIREMENTS AND CORRECTIVE ACTIONS 9

1. Introduction and Scope

1.1 Method Description

1.1.1 This method is applicable to drinking, surface and saline waters, domestic and industrial wastes. The method is suitable for all concentration ranges, however, to avoid large titration volumes, the analyst should use a sample volume that contains less than 25 mg CaCO3.

1.1.2 Calcium and magnesium ions in the sample are sequestered upon the addition of disodium ethylenediamine tetraacetate. The end point of the reaction is detected by means of Eriochrome Black T indicator, which has a red color in the presence of calcium and magnesium and a blue color when the cations are sequestered. The routine reporting limit for this test is 1 mg/L.

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 should be collected in a 500 ml plastic container. Nitric acid is added to lower the pH to <2. Samples should be stored at 4°±2° C. The holding time for Hardness is 6 months from date 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 or 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 Hardness run on March 7,1997 on water would be A030797_HRD_W02.

1.3.3 CLP - Contract Laboratory Program - The USEPA program that contracts with laboratories to provide laboratory services. The term has come to mean a much broader set of methods and deliverables. In context of this SOP, CLP means procedures or operations which are detailed in the CLP contract and which are extended to a broader working definition.

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

1.3.5 PB - Preparation blank - This term is borrowed from CLP. An analysis blank is made up in the same way as calibration standards, without target analytes.

1.3.6 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.

2. Equipment List and Standards

2.1 Apparatus

2.1.1 Standard laboratory titration equipment, i.e., buret, graduated cylinders, erlenmeyer flasks, pipettes.

2.2 Reagents

2.2.1 Buffer solution: Dissolve 1.179g disodium EDTA and 780 mg $MgSO_4.7H_2O$ in 50 ml of DIW. Add this solution to a 250 ml volumetric flask containing 16.9 g NH_4Cl and 143 ml conc NH_4OH with mixing and dilute to the mark with DIW. This reagent should be stored in a tightly stoppered plastic bottle. The solution should be discarded when 1-2 ml added to a sample fails to produce a pH of 10.0±0.1 at the endpoint of titration..

2.2.2 Inhibitors: Laucks does not routinely use inhibitors. If the analyst suspects that the sample may contain significant amounts of interferring metals, they should discuss the use of inhibitors with their supervisor.

2.2.3 Indicator: Mix 0.5 g Eriochrome Black T with 4.5 g hydroxylamine hydrochloride. Dissolve in 100 ml of 95% ethanol.

2.2.4 EDTA titrant: Place 3.723g analytical reagent grade disodium ethylenediamine tetraacetate dihydrate in a 1L volumetric flask and dilute to the mark with DIW. Check the normality of this solution periodically against the standard calcium solution.

2.2.5 Standard calcium solution: Place 1.000g anhydrous calcium carbonate in a 500 ml flask. Add, a little at a time, 1+1 HCl, until all of the $CaCO_3$ has dissolved. Add 200 ml DIW. Boil for a few minutes to expell CO_2. Cool. Add a few drops of methyl red indicator and adjust to the intermediate orange color by adding 3N NH_4OH or 1+1 HCl. Quantitatively transfer to a 1L volumetric flask and dilute to the mark with DIW. One ml of this solution should sequester 1 ml of titrant.

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 The buffer solution contains high concentration of ammonia, which is an irritant. The analyst should use caution around this material.

3.2 Waste Disposal

3.2.1 The wastes generated in this test pose no environmental or health hazard and may be disposed of in the sink.

4. Calibration and Quality Control

4.1 Initial Preparation Blank

4.1.1 At the start of the batch a prep blank is analyzed. The level of hardness in the PB should not exceed 2 mg/L. One prep blank is required for every 20 samples or batch, whichever is less.

4.1.2 Corrective action

4.1.2.1 If the initial PB 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.

4.2 Laboratory Control Sample

4.2.1 Following the prep blank, a laboratory control sample is analyzed. The recovery must be within vendor supplied limits. One LCS must be analyzed for every 20 samples, or every batch, whichever is less.

4.2.1.1 Corrective action

4.2.1.2 If the LCS is out of control, the analysis is terminated. The cause of the failure needs to be identified and rectified. Analysis can only proceed when the LCS is within control limits.

4.3 Sample Duplicate

4.3.1 Criteria

4.3.1.1 Sample duplicates are required. At least one duplicate sample per 10 samples per matrix is required. RPD values are calculated as follows:

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

where:

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

4.3.1.2 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.

4.3.2 Corrective action

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

5.1.1.1 Prep blk
LCS
Sample
Sample duplicate
Up to nine more samples
Sample
Sample duplicate
Up to nine more samples
Start over with Prep blank

5.2 Analytical Operation

5.2.1 Most samples will require no pretreatment.

5.2.2 Place an aliquot of sample in the titration vessel. The size of the aliquot should be chosen so that the titration volume is 15 mls or less, up to a maximum of 50 ml of sample.

5.2.3 Add 1-2 ml of buffer.

5.2.4 Add 1-2 drops of indicator.

5.2.5 Titrate slowly with continous stirring with EDTA titrant until the last reddish tint disappears. The solution should be blue at this point. If the end point is vague, interferrence is suspected, and the use of inhibitors is suggested.

5.2.6 If the hardness is <5 mg/L, an aliquot of 100 ml may be used, with corresponding increases in the amount of buffer and indicator.

5.2.7 The titrant is normalized by titrating a 10.0 ml aliquot of the standard calcium solution to the blue endpoint. The normality is calculated as follows:

$$\text{N of EDTA} = \frac{0.2}{\text{ml of EDTA}}$$

5.3 Compound Quantification

5.3.1 Hardness, mg/L $CaCO_3$ = $\frac{\text{ml of titrant} * N * 50000}{\text{ml of sample}}$

N= normality of EDTA

6. Reports

6.1 Data Packet Organization

6.1.1 Data package consists of the bench sheet 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. A summary of all data entered must be included in the data packet. The routine minimum is a method blank report, and an LCS and a duplicate report.

6.3 Sample Result Reports

6.3.1 Data Qualifying Flags

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

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

Appendix I

QC Summary Table

Laucks Testing Laboratories

Method _130.2__ QA Requirements and Corrective Actions

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Prep Blank	None	Must be ≤2X the reporting limit≤	1/20 or every batch	Reanalyze	QC_DB report
Duplicate % Difference	None	See QC_DB	1/10 or every batch	Reanalyze	QC_DB report
Standard Reference Material (SRM) Recovery	None	Vendor supplied limits	1/20 or every batch	Reanalyze	QC_DB report

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-9110

Determination of Anions by Ion Chromatography by Method 300.0A

Revision history:

Number	Date
4	3/08/97
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2	5/08/96
1.1 (LTL-0030)	5/31/90
1.0 (LTL-0030)	10/19/89

UNCONTROLLED

Written by:: Bill Lundberg — Date: 3/10/97
Bill Lundberg, Inorganics Division Manager

Approved by: Harry Romberg — Date: 3-10-97
Harry Romberg, QC Officer

Approved by: Karen J. Kotz — Date: 3/10/97
Karen Kotz, Laboratory Director

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METHOD 300.0 A QA REQUIREMENTS AND CORRECTIVE ACTIONS 18

1. Introduction and Scope

1.1 Method Description

1.1.1 This method describes the technique used to determine anions in water using Ion Chromatography. The method is capable of being extended to the analysis of anions in soil matrices if a preliminary leaching step is performed to solubilize the anions. Anion determinations may also be performed on the solution resulting from the oxygen bomb decomposition of fuels, solids, oils, and total/organic halogen extracts.

1.1.2 The anions in an aqueous solution are introduced via an autosampler into a flowing eluant stream. This stream is passed through a cation exchange column, which strips the metallic elements from the solution. It then passes through an anion exchange column where the chromatographic separation occurs. The separation column effluent is then passed through an anion micromembrane suppresser where counter ions are exchanged for hydronium ions. This serves to suppress the conductance of the eluant and enhances the conductance of the anionic analytes of interest by forming their complementary acids. Following this exchange the stream is passed through a conductivity detector. The detector output is collected by a data acquisition system, which stores the chromatograms for later calculation and interpretation.

1.1.3 The method is based on EPA 300.0 A, modified as stated in this document. Modifications are marked with ***bold italics***, and listed in the following table:

Method 300.0 Criteria	Laucks Practice
QCS (ICV) must be ± 10%	Vendor limits are used for ICV's but not to exeed 10% of the TV
IPC (CCV) must be ± 10%	Chloride CCV's allowed ± 15%
Sample exceeding high standard must be diluted.	Sulfate samples may be reported to twice the high standard.

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

1.2 Sample Collection, Sample Storage, Holding Times

1.2.1 Samples are collected in 500 or 1000 mL unpreserved polyethylene bottles. Upon receipt, samples are stored 4° ± 2° C.

1.2.2 Analysis holding time is dependent on the anion of interest. Holding time for Nitrate ,Nitrite, and Ortho phosphate analysis is 48 hrs. Holding time for Bromide, Chloride, Fluoride, and Sulfate is 28 days.

1.3 Definition of Terms

CCB - Continuing Calibration Blank - This is the same acronym used in the CLP program. This is a blank which is analyzed immediately after the CCV (almost always after every 10 samples and at the end of the analytical run) during the analysis sequence to determine whether the instrument or system has maintained a stable baseline.

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 - 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.

SRM or LCS - Standard Reference Material or Laboratory Control Sample. This is a material of approximately the same matrix as the samples, containing a known and usually certified amount of target analyte and which is prepared and analyzed in the same manner as a typical sample. This sample is used to demonstrate that the analytical system is in control.

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

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

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

RT window - Retention time window - The +/- value which is applied to the 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.

2. Equipment List and Standards

2.1 Equipment

2.1.1 Ion chromatograph, Dionex model 2000i or equivalent consisting of a dual piston pump, autosampler, either manual or automated injector, guard column, separator column, anion micromembrane suppresser, and a conductivity detector.

2.1.2 Guard column, Dionex AG4A or equivalent

2.1.3 Chromatographic column, Dionex AS4A or equivalent

2.1.4 Anion Micro Membrane Suppresser, Dionex AMMS-1 or equivalent

2.1.5 Electronic data collection device, Ezchrom data acquisition system or equivalent, or other applicable data collection system.

2.2 Standards and Solutions

2.2.1 Calibration Standard/Spiking Solution.

2.2.1.1 A 100 mL calibration standard/spiking solution is prepared as stated in the following table from vendor supplied primary standards. The primary standards are stored in the standards refrigerator at 4 °C. They are replaced annually. Upon receipt the bottles are logged into the standard logbook and are QC'd.

Analyte	Stock Conc. (mg/L)	Volume of Stock (µls)	Final Conc. (mg/L)
F	1000	500	5
Cl	1000	1000	10
NO_2 as N	100	2000	2
Br	1000	500	5
NO_3 as N	1000	500	5
PO4 as P	50	10000	5
SO_4	1000	2000	20

The above solution expires after 3 months. It is stored at room temperature.
The routine calibration solution does not contain Br, and PO4. These analytes are added when required.

2.2.1.2 Spiking - Samples are spiked by adding 100µl of solution prepared in 2.2.1.1 to an aliquot of sample and diluting to 500 µl. The volume of sample, the volume of spike, and the spike reference number are recorded in the pretreatment field of the batch summary.

2.2.2 ICV Solutions.

2.2.2.1 An ICV mix for fluoride, chloride, nitrate, phosphate and sulfate is vendor purchased.

2.2.2.2 A 1. mg/L nitrite ICV solution is prepared by diluting 100 µL of a 1000 mg/L vendor supplied solution to 100 mls in a volumetric flask. This solution is prepared weekly.

2.2.2.3 A 1000 mg/L primary bromide ICV solution is prepared by diluting 0.1288 g of desiccated NaBr to a final volume of 100 mL. This solution is prepared annually Desiccated NaBr is prepared by first drying the salt in a 105°C ± 5°C drying oven for one hour. The salt is then cooled to room temperature in a desiccator.

2.2.2.4 A 5. mg/L working bromide ICV is prepared by diluting 250 µL of 2.2.2.3 to a final volume of 50 mL. This solution is prepared fresh daily.

2.2.3 Eluant Solution.

2.2.3.1 The eluant is 1.7 mM sodium carbonate and 1.8 mM sodium bicarbonate. The eluant is prepared by dissolving 0.290 g sodium bicarbonate and 0.380 g sodium carbonate in DIW and diluting to a final volume of 2 L.

3. Safety precautions and Waste Disposal

3.1 Safety Precautions

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

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

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

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

3.2 Waste Disposal

3.2.1 There is no significant waste generated from this procedure. Any remaining liquids are disposed of by flushing down the sink. Solid material may be disposed of in the garbage.

4. Calibration and Quality Control

4.1 Method Detection Limit Study

4.1.1 Prior to the analysis of any samples, it is necessary to establish method detection limits. This procedure is fully described in the Laucks SOP on the determination of detection limits. This SOP can be found in the SOP manual. Briefly, it involves the analysis of 7 replicate samples spiked at a concentration near the anticipated method detection limit. A Student's T-test is then applied to these measured values to calculate the MDL.

4.1.2 Per method 300.0, MDL studies for the ion chromatograph must be determined at a minimum of every six months, with each new analyst, or whenever there is a significant change in system response.

4.2 Initial Multi-Point Calibration

4.2.1 A calibration curve consists of four points. All calibration standards are diluted in sample vials at the instrument just prior to analysis as stated in the following table:

Standard Number	Volume Standard	Volume DIW	Total Volume
1	500 µl	0 µl	500 µl
2	250 µl	250 µl	500 µl
3	100 µl	400 µl	500 µl
4	25 µl	475 µl	500 µl

4.2.2 Samples that exceed the high standard must be diluted, ***with the exception of sulfate, which is reported to twice the high standard.*** The extended linearity of sulfate is supported by the vendor supplied ICV solution. The vendor supplied ICV has a sulfate concentration that can be accurately determined at instrument concentrations greater than twice the high sulfate standard.

4.2.3 In the event that the vendor supplied ICV has a sulfate value of less than 40. mg/L, a 50. mg/L sulfate solution will be run at a minimum of once per calibration. This solution must return a value of ± 10% to validate the extended sulfate linearity.

4.2.4 A multi-point calibration of the system is not required with every analytical run.

4.2.5 If the previous calibration is verified by an ICV, the calibration is considered valid and the instrument does not require re-calibration.

4.2.6 A curve is not valid for a period of greater than six months.

4.2.7 Criteria

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

4.2.8 Corrective action

4.2.8.1 If the criteria are not met, the instrument must be recalibrated. No data can be reported for an analyte that does not have a valid calibration.

4.3 Initial Calibration Verification

4.3.1 At the start of each analytical run, analyze a standard from a source other than that from which the calibration material was obtained.

4.3.2 Criteria

4.3.2.1 ***The calculated concentration of the ICV for fluoride, chloride, nitrate, phosphate and sulfate must be within the limits supplied by the manufacturer*** or not to exceed ±10% of the true value. The calculated concentration of the ICV for bromide and nitrite should not exceed 90%-110% of the true value.

4.3.3 Corrective action

4.3.3.1 If the ICV criteria are not met, no samples can be analyzed. Perform system maintenance and re-check the ICV. If the criteria still cannot be met, the system must be recalibrated. No data can be reported for an analyte that does not have a valid ICV.

4.4 Initial Calibration Blank

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

4.4.2 Corrective action

4.4.2.1 If the ICB 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. No data can be reported for an analyte that does not have a valid ICB.

4.5 Continuing Calibration Verification (CCV) and Blank (CCB)

4.5.1 A mid-range calibration standard is analyzed at a minimum of once every 10 sample injections. Immediately following the CCV, a blank solution is analyzed. In addition, this standard and blank must be the last samples analyzed in the run.

4.5.2 Criteria

4.5.2.1 The CCV must fall within ± 10% of the true value, ***within ± 15% for chloride***.

4.5.2.2 The levels of target analytes in the CCB should not exceed the reporting limit.

4.5.3 Corrective action

4.5.3.1 If CCV limits are exceeded, check calculations or perform instrument maintenance. Recalibrate and reanalyze. No sample results may be reported that are not bracketed by a successful ICV and a CCV which is in control or by preceding and following CCVs which are within limits.

4.5.3.2 If the 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, whichever is more frequent. Any analyte response above the reporting limit is reported. For the purpose of water analyses, the ICB, CCB's, and the method blank are the same. Method blank control limits are that contamination should not exceed the reporting limit for waters. Method blanks should not exceed twice the reporting limit for soil leaches, oxygen bomb extracts, and total/organic halogen extracts.

4.6.2 Corrective action

4.6.2.1 Corrective action may necessitate re-preparation and re-analysis of the sample set. For example if an analyte were found in the blank but not in any of the associated samples then sample group may not require re-analysis. In addition, if sample levels exceed 10 times the blank, the level of contamination may be considered insignificant. In any case, if re-preparation and re-analysis is not being undertaken, the analyst must first discuss the issue with the Quality 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 Samples are spiked by adding 100µl of calibration/spiking solution to an aliquot of sample and diluting to 500 µl. The volume of sample, the volume of spike, and the spike reference number are recorded in the pretreatment field of the batch summary.

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

sample is used to evaluate the matrix effect of the sample upon recovery of the analytes. The recovery of spike analytes is calculated as follows:

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

where: SS = concentration in spiked sample
S = native concentration in unspiked sample
SA = spiked added, the amount of spiking material actually added to the sample calculated on the sample basis

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

4.7.4 Corrective action

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

4.8 Matrix Spike Duplicate

4.8.1 The compound recovery criteria are identical to those for the matrix spike sample. In addition, the matrix spike duplicate is used measure method precision. This is done by computing the relative percent difference (RPD) between the matrix spike and matrix spike duplicate recovery values. This calculation is as follows:

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

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

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

4.9 Sample Duplicate

4.9.1 Criteria

4.9.1.1 Sample duplicates are required only when the client requests or when CLP practices are employed. At least one duplicate sample per 20 samples per matrix is required when matrix spikes/ matrix dups 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

4.9.1.2 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.

4.9.2 Corrective action

4.9.2.1 If a trend in out of control RPD values is observed, the methods used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable reproducibility. Generally, if recoveries are in control and no analyte of interest was detected in any of the samples, no immediate action will be taken on that sample set. If integrity of reported sample values is in doubt, re-analysis may be called for. Corrective actions should be discussed with the Quality Control Officer.

5. Operation procedures

5.1 Sample Analysis

5.1.1 Analysis tools and observations

5.1.1.1 If a discrete bottle number is referenced on the sample container, this information must be recorded in the pretreatment field of the bath summary.

5.1.1.2 Samples that appear overly turbid, or that have suspended or floating particles need to be filtered through a 0.45 µm syringe filter prior to analysis. If this procedure is followed, it should be documented in the pretreatment field of the batch summary

5.1.1.3 If conductivity or TDS data is available, this can be used as a tool in deciding what dilutions are appropriate.

5.1.1.4 Poor resolution between chloride and nitrite, phosphate and sulfate, and nitrate and bromide in standards or CCV's is an indication that column needs to be cleaned. If after cleaning the CCV does not resolve back to baseline between the two peaks of interest, the column has lost enough theoretical plates to render it unsuitable for analysis of those ions.

5.1.1.5 An abnormally shaped sulfate peak may indicate a sample with a high phosphate concentration. The chromatogram should be compared to one run at a greater dilution to see if peak resolution can be obtained.

5.1.1.6 The expected baseline conductivity for the system is 17.5 to 19.0 µs.

5.1.1.7 The expected back pressure of the system is 750 to 1200 psi. When the upper limit is approached the analyst should replace the eluant filter, replace the bed supports, clean the column, or reduce the flow rate. It is acceptable to reduce the flow down to 1.2 ml/min.

5.1.1.8 Other anomalies should be discussed with the departmental supervisor in order to determine appropriate corrective actions.

5.2 Analysis sequence

Calibration standards (from high to low concentration)
ICV Solutions necessary for that analytical run
ICB
10 or fewer samples
midrange standard (CCV)
DIW (CCB)
additional samples (not more than 10)
additional CCV's and CCB's
...
...
must close with CCV and CCB

5.3 Instrumental Conditions

5.3.1 System Conditions

- Eluant flow is between 1.0 and 2.0 mL/min.
- Injection volume is 50 µL.
- Temperature correction factor is 1.7.
- Detector range is 30 µS.
- Analysis time is between 10 and 13 minutes.

5.4 Analytical Operation

5.4.1 Analytical procedure for liquid matrixes

5.4.1.1 A set of samples is chosen to be analyzed. Consideration should be given to holding times, due dates, and analytes of interest when choosing the sample set for a run.

5.4.1.2 A run log is constructed for the sample set. This includes the appropriate dilutions for the given samples.

5.4.1.3 The run log is programmed into the integrator or equivalent device (along with the sample dilutions), the run and sample counters are reset, and the end run statement is input.

5.4.1.4 An appropriate number of 500 µL sample vials are rinsed and placed into the auto sampler trays.

5.4.1.5 500 µL of standards, samples and QC events are placed into the sample vials.

5.4.1.6 Dilutions up to 50x can be done directly in the sample vials, greater dilutions will require an external dilution in a Solo cup or a combination of external and sample cup dilutions.

5.4.1.7 Samples that appear turbid or that have suspended particles should be filtered through a 0.45 µm syringe filter prior placing in sample vials and/or dilution. If a sample is pre-filtered, it must be documented in the pretreatment field of the batch summary.

5.4.1.8 Sample vials are capped with filter caps and placed in the auto sampler.

5.4.1.9 The "Inject A" button on the integrator and the remote button on the automation interface are pushed simultaneously to begin analysis.

5.4.2 Analytical procedure for soil or sludge matrixes.

5.4.2.1 The soil/sludge is leached with DIW by transferring 10. g to a tared 250 mL digest bottle, adding 100 mL DIW and placing on the shaker for 15 minutes or more at a shaking rate sufficient to suspend the majority of the solids. The solids are then allowed to settle and an aliquot of the supernatant is filtered through a 0.45 µm syringe filter prior to use. The leaching and filtration of the sample must be recorded in the pretreatment field of the batch summary.

5.4.2.2 A 100 mL aliquot of DIW is also shaken in a 250 mL digest bottle to be used as a method blank.

5.4.2.3 The resulting supernatant is treated as a water sample.

5.4.2.4 A total solids determination is made on all soil/sludge samples and the results are reported on a dry weight basis.

5.5 Compound Quantification

5.5.1 The data aquisition system generates a report with the chromatogram of the sample, the date analyzed, the sample identification, the method used to quantify the sample, the sample dilution, and the calculated concentration of the sample corrected for dilution.

5.5.2 For soil/sludge samples, the mg/L integrator value is converted to mg/Kg DB by the following equation

Soil: $$Cs = \frac{Ci * Vs * 100}{Ws\ Ts}$$

Cs = Sample concentration (mg/kg DB)
Ci = Concentration in sample injected (mg/L)
Vs = Volume of extract (mL)
Ws = Weight of sample extracted (gm)
TS = Total solids (%)

5.5.3 Samples that exceed the high standard must be diluted, ***with the exception of sulfate, which is reported to twice the high standard.*** The extended linearity of sulfate is supported by the vendor supplied ICV solution, which has a sulfate concentration which is greater than twice the high sulfate standard.

6. Reports

6.1 Sample Results and Quality Control Reports

6.1.1 Data packages shall consist of the sample results summary, the chromatograms for samples in the workorder, and a copy of the QC summary report form. These include the method blank analysis, matrix spike results, duplicate results, and the Standard Reference Material (SRM) reports. A copy of the QC summary report form must be included in order to indicate that the QC was evaluated for analytical consistency.

6.2 Data Qualifying Flags

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

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

Appendix I

Laucks Testing Laboratories
Method 300.0 A QA Requirements and Corrective Actions

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Initial Calibration	A minimum three point curve, that brackets the sample concentrations and a blank.	A four point curve, that brackets the sample concentrations with the exception of sulfate, which is reported to twice the high standard.	As instrument performance dictates, or when any change is made in the analytical system.	Fix the problem and recalibrate.	In the raw data package for all validatable packages and referenced in all other data.
Initial Calibration Verification / SRM	Not to exceed ±10% of the true value.	Nitrite and bromide to ±10% of the true value. ***Fluoride, chloride, nitrate, phosphate and sulfate values are vendor supplied*** but must not exceed ±10% of the true value.	Once per analytical run, or 1/20.	Reanalyze, if reanalysis fails recalibrate and or perform instrument maintenance. No data can be reported if ICV fails.	In the raw data package, and in the quality control data base.
Initial Calibration Blank	Value not to exceed the MDL.	Value not to exceed the Reporting Limit.	Immediately following the ICV.	Reanalyze. No data can be reported if ICB fails.	In the raw data package, and in the quality control data base.
Continuing Calibration Verification	Not to exceed ±10% of the true value.	Not to exceed ±10% of the true value. ***Not to exceed ±15% for chloride.***	At a minimum, after every 10 analytical samples and as the penultimate injection.	Samples which are not bracketed by in control CCV's must be reanalyzed.	In the raw data package.
Continuing Calibration Blank	Value not to exceed the MDL.	Value not to exceed the Reporting Limit.	Immediately following a CCV's.	Samples which are not bracketed by in control CCB's must be reanalyzed.	In the raw data package.

Laucks Testing Laboratories
Method 300.0 A QA Requirements and Corrective Actions

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Method Blank	Value not to exceed the MDL.	ICB and method blank are the same QA event for waters. Not to exceed 2x the reporting limit for non-water matrixes.	Run at a minimum of 1 per 20 samples and as dictated by non-water matrixes.	If sample concentration exceed 10x the contamination level, no action required. Samples with contaminated Method Blanks must be re-extracted.	In the raw data package, and in the quality control data base.
Matrix Spike Recovery	Not to exceed ±20% of the true value or x ± 3S. Once every 10 samples.	To meet or exceed criteria in the current lab control limits catalog or in quality control data base.	Once every 20 samples, or per client request, whichever is more frequent.	Review data, consult QC officer, some conditions may require reanalysis.	Narrative comment in report in quality control data base, and in raw data package.
MS/MSD RPD	NA	To meet or exceed criteria in the current lab control limits catalog or in quality control data base.	Once every 20 samples, or per client request, whichever is more frequent.	Review data, consult QC officer, some conditions may require reanalysis.	Narrative comment in report in quality control data base, and in raw data package
Duplicate/ % Difference	NA	To meet or exceed criteria in the current lab control limits catalog or in quality control data base.	Once every 20 samples, or per client request, whichever is more frequent.	Review data, consult QC officer, some conditions may require reanalysis.	Narrative comment in report in quality control data base, and in raw data package.

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #: LTL-9113

Title: **pH in Soil and Waste Samples Using SW846 Method 9045C**

Revision history:

Number	Date
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Written by: Bill Lundberg, Inorganic Supervisor — Date: 1/16/97

Reviewed by: Harry Romberg, QA Officer — Date: 1-16-97

Approved by: Karen Kotz, Lab Director — Date: 1/16/97

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ıtroduction and Scope

/lethod Description

This method is an electrometric procedure for the determination of pH in soils and waste les.

The sample is mixed with deionized water (DIW). The pH of the solution is then ıred with a pH meter.

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

nterferences:

Samples with very low or very high pH may give incorrect readings on the meter. For es[illegible]n a true pH of >10, which is not uncommon for some ash and other waste materials, easured pH may be incorrectly low. This error can be minimized by using a low-sodium- electrode. Strong acid solutions, with a true value of <1, an uncommon condition, may ncorrectly high measurements.

Temperature fluctuations will cause measurement errors. Most pH electrodes are ated to 25°C. Whereas Laucks does use a temperature correction probe with the pH meter, icant differences in pH can affect the solubility and apparent pH characteristics of the on itself. Therefore, measurements should be made near the appropriate temperature by ing the sample and diluent (DIW) to warm to room temperature prior to analysis.

Errors can occur when the pH electrode becomes coated. If this occurs, the electrode must roughly clean and re-buffered prior to measuring the pH of subsequent samples.

ample Collection. Sample Storage, Holding Times

There is no official holding time for the analysis of soil pH. The holding time for aqueous es is to analyze immediately. Soil should not demonstrate significant pH fluctuations if erated and analyzed within a reasonable time but Laucks has chosen to analyze soils es as soon as is reasonable after collection, not to exceed 7 days from collection.

1.4 Definition of Terms

1.4.1 This section defines terms and acronyms as they are used in this SOP. Other terms, such as MS/MSD or method blank, are not defined here since it is assumed that the user of this SOP already understands their more general meaning.

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

1.4.1.2 **Sequence** - A set of sample extracts and standard solutions presented to an instrument in a chronologically continuous group.

2. Equipment List, Reagents and Standards

2.1 Apparatus

pH meter with means for temperature correction (temperature correction probe)
Glass electrode
50 ml. beaker
Analytical balance capable of weighing to 0.1 g.

2.2 Standards

pH 4 buffer
pH 7 buffer
pH 10 buffer

2.3 Reagents

Deionized water (DIW)

3. Safety precautions and Waste Disposal

3.1 Safety Precautions

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

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

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

3.2 Waste Disposal

3.2.1 There should be no significant waste associated with this procedure which cannot be disposed of in the laboratory sink and/or garbage.

4. Calibration and Quality Control

4.1 Calibration

4.1.1 The pH meter must be calibrated with at least two pH buffers. One is the pH 7 buffer and the other depends upon the samples. If high pH is expected, the second buffer should be at pH 10 or higher. If low pH is expected, the second buffer should be the pH 4 buffer. High pH samples should not be analyzed from a low pH calibration and visa versa.

4.2 Sample Duplicate

4.2.1 Criteria

4.2.1.1 At least one duplicate sample per 10 samples per matrix is required. RPD values are not currently calculated for this analysis. The control limit is considered to be 0.5 pH units.

4.2.2 Corrective action

4.2.2.1 If an out-of-control duplicate pair is observed, the methods used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable reproducibility. If integrity of reported sample values is in doubt, re-analysis may be called for. Corrective actions should be discussed with the Quality Control Officer.

5. Operation procedures

5.1 Sample preparation and pH measurement of soils

5.1.1 To 20 grams of soil in a 50 ml beaker, add 20 mls of DIW and stir the suspension for 5 minutes. This may also be done with a magnetic stirring bar.

5.1.2 Allow the suspension to stand for about 1 additional hour with no agitation in order to allow most of the suspended particulates to settle out.

5.1.3 Immerse the electrode (including the temperature correction probe) just far enough into the solution to establish good electrical contact. It should not come into contact with the soil itself.

5.1.4 The electrodes should be thoroughly rinsed between samples.

5.1.5 Report the results as "soil pH measured in water @ ____ °C."

5.2 Sample preparation and pH measurement of waste material:

5.2.1 To 20 grams of waste material in a 50 ml beaker, add 20 mls of DIW and stir the suspension for 5 minutes. This may also be done with a magnetic stirring bar.

5.2.2 Allow the suspension to stand for about 15 additional minutes with no agitation in order to allow most of the suspended particulates to settle out.

Note: If the waste is hygroscopic and absorbs all the DIW, begin the test again using 20 g of waste and 40 ml of DIW.

Note: If the supernatant is multiphasic, decant the oily phase and measure the pH of the aqueous phase.

5.2.3 Immerse the electrode (including the temperature correction probe) just far enough into the solution to establish good electrical contact. It should not come into contact with the waste material itself.

5.2.4 The electrodes should be thoroughly rinsed between samples.

5.2.5 Report the results as "waste pH measured in water @ _____ °C.

6. Reports

6.1 Data Packet Organization

6.1.1 The data package for this analysis consists of the data sheet and a quality control database (QC_DB) report form.

6.2 Quality Control Reports

6.2.1 All results for quality control tests are entered into the lab data base using the QC_DB program. The report form must be filled out in order to indicate that the duplicate QC had been performed and met the criteria specified in this document.

Appendix I

QC Summary Table

Laucks Testing Laboratories

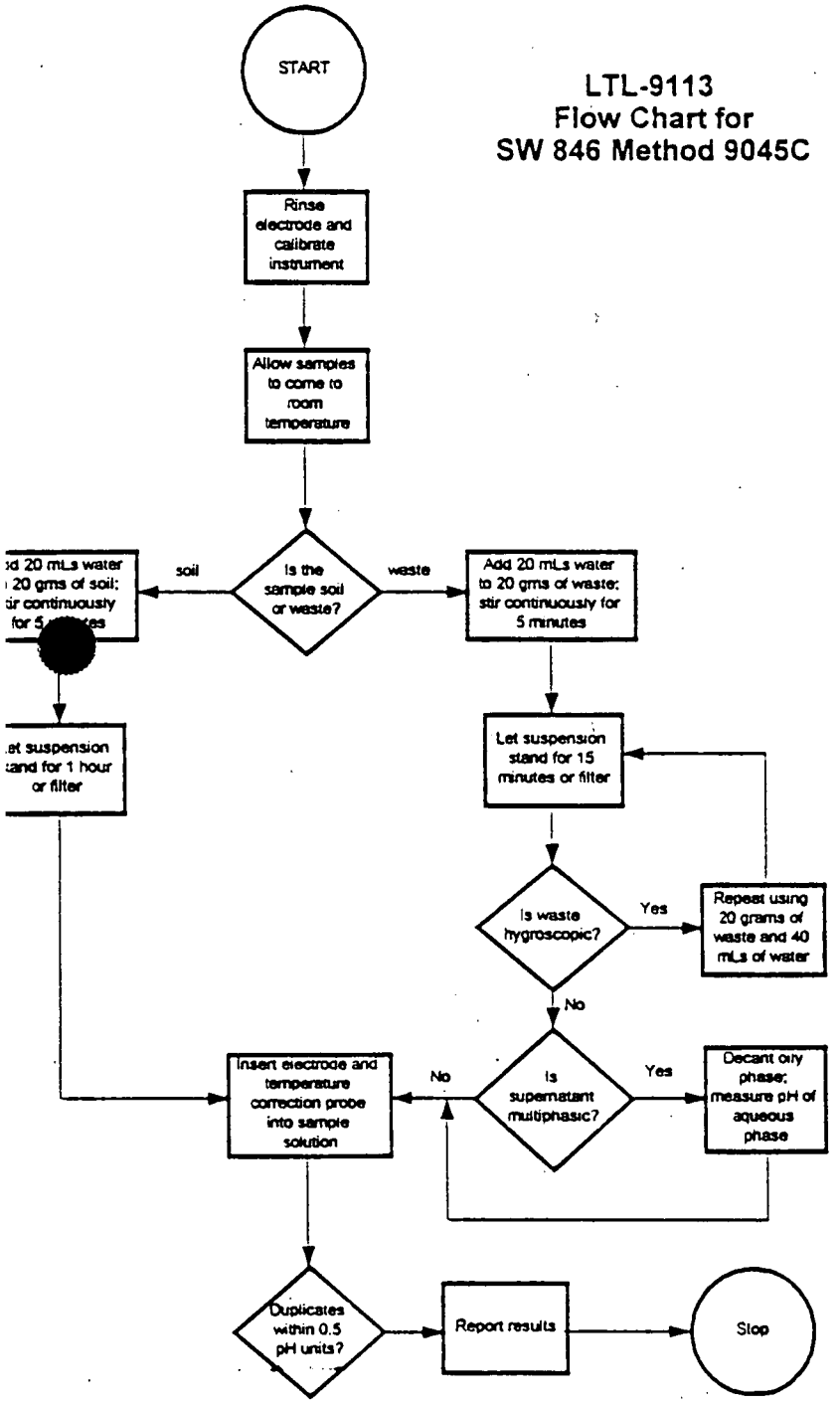
Method SW 9045C QA Requirements and Corrective Actions

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Initial Calibration	Buffer pH meter to bracket sample values	Buffer pH meter between pH 7 & 4 or 10 before analyses (to bracket sample values)	Once before analyzing any samples or QC	None	None
Duplicate % Difference	N/A	within 0.5 pH units of one another	10% or once every 10 samples.	Correct problem Consult QC Officer	QC_DB report form with appropriate commentary

Appendix II

Flow Chart

LTL-9113
Flow Chart for
SW 846 Method 9045C



LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP No. **LTL-9116**

Previous SOP No. **LX-0049**

Title: **The Determination of Total Organic Carbon in Soil/Sediment Samples Using Modified SW846 Method 9060**

Rev: 1

UNCONTROLLED

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.

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #: ~~LX-0049~~ 12/22/95
LTL-9116

itle: **The Determination of Total Organic Carbon in Soil/Sediment Samples Using Modified SW846 Method 9060**

evision history:

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evised by: Harry Romberg, Quality Control Officer — Date: 10-4-94

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eviewed by: Mike Nelson, Technical Director — Date: 9/30/94

pproved by: John Buerger, Laboratory Director — Date: 10/4/94

Introduction and Scope

1. Method Description

1.1. This method is used to determine the concentration of total organic carbon in a soil or sediment sample. Inorganic carbon (carbonates) are removed by treatment of the homogenized sample with 1:1 nitric acid. The treated sample is then combusted in a furnace and the carbon dioxide evolved as a combustion product is measured using an infrared detector.

1.2. This method is based on SW846 method 9060, modified to accommodate the analysis of soils and sediments.

1.3. Assuming a 1:5 dilution factor, using 10 to 100 mg of diluted sample, the stated calibration curve allows for a concentration range of 0.1 to 2.0% carbon in the sample. This range can be extended in either direction by adjusting the sample dilution.

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

2. Sample Collection, Sample Storage, Holding Times

2.1. Samples can be collected in glass or plastic containers. A minimum sample size of 50 grams is recommended.

2.2. Samples should be stored at 4°C ± 2°C. The holding time for soil samples is not established, but the holding time for water samples is 28 days from sample collection. For lack of other official guidance, 28 days from sample collection is used for the analytical holding time.

3. Definition of Terms

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.

3.2. CCV - Continuing calibration verification. - This is the same acronym used in the CLP program. This is a standard analyzed at some prescribed frequency (almost always after every

10 samples) during the analysis sequence to determine whether the instrument or system has remained in calibration.

1.3.3. **CLP** - Contract Laboratory Program - The USEPA program that contracts with laboratories to provide laboratory services. The term has come to mean a much broader set of methods and deliverables. In context of this SOP, CLP means procedures or operations which are detailed in the CLP contract and which are extended to a broader working definition.

1.3.4. **Corr Coef, CC** - 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

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

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

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

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

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

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

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

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

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

3.14. **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

1. Equipment

1.1. TOC analyzer, Dohrman DC-80 or equivalent
1.2. Dohrman 183 boat sampler or equivalent
1.3. Analytical Balance: 0.1 mg accuracy
1.4. Desiccator
1.5. Platinum combustion boats
1.6. Drying oven set at 70°C

2. Reagents and Standards:

2.1. Nitric acid (HNO_3) Prepare by diluting 50 ml concentrated HNO_3 to 100 ml with DIW.
2.2. ASTM Type II water
2.3. Potassium hydrogen phthalate (KHP) Stock Solution: 4000 mg/L carbon: Dissolve 1700 g ±1 mg KHP in a 200 ml volumetric flask. Calibration solutions are prepared from the stock dilution.

2.3.1. Prepare solutions at 500, 1000, 1500 and 2000 mg/L as follows. Prepare a 2000 mg/L solution by pipetting 50 ml of the 4000 mg/L stock solution to a 100 ml volumetric flask and diluting to 100 ml with DIW. In a similar manner, prepare a 1500 mg/L standard by diluting 37.5 ml of the 4000 mg/L solution to 100 ml, a 1000 mg/L solution by diluting 25 ml of the 4000 mg/L solution to 100 ml, and a 500 mg/L solution by diluting 12.5 ml of the 4000 mg/L solution to 100 ml.

2.2.4. Glucose ICV solution- 1000 mg/L carbon as C- Dissolve .250 grams of glucose in 100. mls. of deionized water.

2.2.5. Sand, which has been disk pulverized to pass at least 80 mesh, is placed in a shallow ceramic evaporating dish and ignited in the muffle furnace at 700°C for at least 2 hours.

2.2.6. Reference material is Buffalo River Sediment, NIST 2704 or equivalent.

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. The combustion furnace is heated to approximately 950°C. Care should be taken not to burn oneself when in the proximity of the furnace or objects which have been recently removed from the furnace.

3.2. Waste Disposal

3.2.1. There is no significant waste generated from this procedure. Any remaining liquids are disposed of by flushing down the sink. Solid material (ash, soil, etc.) may be disposed of in the garbage.

Calibration and Quality Control

1. Method Detection Limit Study

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.

2. Initial Multi-Point Calibration

2.1. The initial, multi-point calibration should be done for every analytical run.

2.2. A small piece of quartz wool is placed into the boat. This should be replaced daily. A 40 µl aliquot of the 2000 mg/L standard is injected onto the quartz wool.

2.3. Press the "start" button, and slide the boat smoothly into the furnace at about two inches per second.

2.4. Repeat this procedure with each of the other calibration standards (1500, 1000, and 500 mg/L). In addition, analyze a calibration blank (DIW) just after the last calibration standard. Report this blank as a sample with no dilution.

2.5. Calibration factor

2.6. A calibration factor is calculated for each standard and the mean calibration factor is used for computing sample results.

$$CF = \frac{(\mu L * C)}{(R * 1000)}$$

ıere:
CF = calibration factor (µg C per response unit)
µl = µl standard analyzed
C = concentration of standard analyzed (mg/L)
R = instrument response

2.7. Criteria

2.7.1 The RSD of the calibration factors for the initial calibration must be 10%.

..8. Corrective action

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

. Initial Calibration Verification

.1. Criteria

.1.1. The 1000 mg/L C (.25% glucose) ICV material is injected immediately following initial calibration. It should read ±10% of the "true" value (between 900 and 1100 mg/L).

.2. Corrective Action

alysis of actual samples should not be started unless calibration of the instrument has been ified. If the ICV exceeds limits, the first step might be to make up new glucose solution, as is probably the least stable solution. If that does not prove to be the cause of the failed ibration, new KHP solutions should be made up and/or instrument cleaning and maintenance uld be undertaken. **No sample values should be reported from any run where the ibration has not been adequately verified.**

. Initial Calibration Blank

.1. No initial calibration blank is analyzed with this analysis. A method (sand) blank is analyzed and all sample values are blank corrected. See the method blank part of this method.

. Method Blanks

.1. This method does not utilize a method blank, as normally defined. This is because all samples are pre-diluted, in a non-standard way, using the diluent blank. Since this material is known to exhibit a background level of carbon, the variable contribution of this background to each diluted sample is subtracted out to compute the "true" native organic carbon concentration.

.2. Since there is not a "method blank" in this method, there is no reporting requirement.

6. Field rinsates, field blanks

6.1. Criteria

6.1.1. Field rinsates and blanks are analyzed only upon client request and are not a routine part of the method. These aqueous samples are analyzed in a manner similar to calibration standards. That is, 40 µl of the aqueous sample is injected onto quartz wool in a combustion boat and ignited. The measured TOC value is computed assuming a 40 mg sample size with no dilution.

6.1.2. The TOC content cannot exceed the reporting limit.

6.2. Corrective action

6.2.1. Reanalyze the affected field QC sample. Notify the client project manager immediately since all samples from the set are potentially adversely affected.

7. Diluent Blank

7.1. Before any samples are analyzed it is necessary to analyze at least three 20 to 50 mg aliquots of the diluent sand. The actual aliquot size is unimportant; the only necessity being that the actual weight of the aliquot is known and recorded. The "sand correction" is calculated as follows:

$$Corr = \frac{R}{W}$$

ıere:
Corr = correction (response/mg)
R = instrument response
W = weight (mg) sand burned

8. Method Detection Limit Standard

9. No method detection limit standard is utilized for evaluation in this analysis. However, the low standard is very near the reporting limit and must meet the calibration criteria discussed in the calibration section of this method.

). Continuing Calibration Verification (CCV)

).1. Criteria

).1.1. A mid-range (1000 mg/l) calibration standard is analyzed after every 10 samples. In addition, this standard must be the last sample analyzed in the run. The %D (difference) of the CCV is calculated as follows and must not exceed 10% of the true value.

$$\%D = \frac{(R - R_i)}{R_i} * 100$$

ere:
%D = % difference
R_i = the raw response of the midpoint calibration standard in the initial calibration curve
R = the raw response of this standard when analyzed as the CCV

0.1.2. Note: The value is automatically calculated in the spreadsheet commonly used by the laboratory to calculate results.

0.2. Corrective action

0.2.1. 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.

1. Matrix Spike

1.1. Criteria

1.1.1. A sample is chosen at random from the samples to be analyzed, unless a client has specifically requested that a certain sample be spiked. An appropriate aliquot of the 1000 mg/L calibration solution is added to this sample prior to the combustion step. This addition should be at least double the instrument response. As the purpose of the matrix spike is to test the system under "typical" conditions, the analyst may avoid selecting the most difficult sample of the batch for spiking. It is not always required that a matrix spike analysis be performed with each preparation/analysis batch, however, the minimum frequency for MS analysis is 1 each per 20 samples per matrix. This matrix spike sample is used to evaluate the matrix effect of the sample upon recovery of TOC. The TOC recovery is calculated as follows:

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

ere:
SS = concentration in spiked sample
S = native concentration in unspiked sample
S_{true} = actual ("true") concentration of spike added

1.1.2. The recovery control limits are 50 - 150%, unless otherwise defined in the QC database and Control Limits Catalog.

1.2. Corrective action

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

2. Sample Duplicate

2.1. Criteria

2.1.1. At least one duplicate sample per 20 samples per matrix is required. The same sample selected as the matrix spike sample is also selected for the duplicate analysis unless the client has specifically requested that another sample be used.

2.1.2. RPD values are calculated as follows:

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

ere:
= measured concentration in the initial analysis
= measured concentration in the duplicate analysis

2.1.3. The RPD control limit is 20%, unless otherwise defined in the QC database and Control Limits Catalog.

12.2. Corrective Action

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

13. Standard Reference Material

13.1. Criteria

13.1.1. Reference material is Buffalo River Sediment, NIST 2704 or equivalent.

13.1.2. The standard reference material (SRM) must be analyzed at a frequency of once per 20 samples or per analysis batch, whichever is more frequent.

13.1.3. The measured TOC value must be within the limits established by the SRM vendor. Alternatively, these limits may be unachievably narrow and may be internally defined by the QC Officer, as is the case with NIST 2704. Currently accepted limits are defined in the laboratory QC database, QC_DB.

13.2. Corrective Action

13.2.1. Appropriate corrective actions may include any or all of the following: check calculations, perform instrument maintenance, recalibrate the instrument, reanalyze all affected samples. Contrary actions must be accepted by the Quality Control Officer before data may be reported.

13.2.2. In any case, the SRM results must be recorded in the QC_DB database and any corrective actions noted.

Operation procedures

.. Instrumental Conditions

..1. The instrument with boat inlet is assembled according to the manufacturer's instructions.

..2. A gas flow of 200 ml/min O_2 is set for the furnace.

:. Analysis sequence

ibration standards (including calibration blank)
nd blanks (at least 1 per batch in triplicate)
ference Material
or fewer samples (may include QC)
ipoint calibration standard (CCV)
ibration blank (CCB)
iitional samples (no more than 10)
iitional CCVs and CCBs

st close with CCV and CCB

. Sample Analysis

.1. Sample preparation

.1.1. It is critical that each sample be thoroughly homogenized before a subsample is taken for analysis. If the sample is already finely divided with no large pieces, a homogeneous sample of 5 to 10 grams is dried in a 70°C oven. If samples contain larger pieces or if they in any way may compromise the analyst's ability to obtain a representative sample, the analyst should utilize sample size of at least 20 grams.

.1.2. If the sample is already finely divided as described above, the dried sample, if it has formed clumps in the drying process, may be ground with a mortar and pestle to pass an 80 mesh sieve.

.1.3. If the sample is sandy, rocky, or as described above, required a larger sample size, it should be ground using the disk pulverizer.

.1.4. Following the grinding step, mix the ground sample thoroughly and combine a weighed representative portion with clean diluent sand in a ratio of 1 part by weight sample to 4 parts by weight of sand. Record the weights of both sample and sand used. The mixture is placed in a snap-cap vial and tumbled for 90 minutes in the TCLP extractor. Dried, pulverized samples should be held in a desiccator.

.2. TS_{105} determination

.2.1. The moisture loss between 70°C and 105°C is considered to be minimal in almost ;ases. In fact, it is considered as likely that TOC losses will occur as that moisture will affect determinations. However, it is necessary to demonstrate this for at least some sample on each ject. The total solids between 70°C and 105°C should be determined on several (at least 10%) he initial sample batch. If the solids are determined to be ≥90%, it is not necessary to perform more TS_{105} determinations for that entire **project**. It is, however, necessary to insert a copy he form for the TS_{105} determinations with each data packet. This form is presented in pendix III. If any of the TS_{105} determinations indicate solids of <90%, these determination st be made for all subsequent samples in the project and used in the quantification of the TOC.

.2.2. Take the remaining ground sample, having been dried at 70°C, and dry it again at 105°C. Calculate the total solids equivalent of the prepared sample as follows

$$TS_{105} = \frac{(W_{105} - tare)}{(W_{70} - tare)} * 100$$

ere:
W_{105} = gross weight of sample and weighing vessel after 105°C
W_{70} = gross weight of sample and weighing vessel after 70°C
tare = weight of empty weighing vessel
TS_{105} = total solids

.2.3. The TS_{105} is used in the calculation of TOC concentration.

.2.4. Instrument startup

.2.5. The furnace is turned on and the temperature allowed to stabilize. It takes approximately 90 minutes for this to happen.

.2.6. The instrument is set to the 40 µl range TOC mode.

.2.7. Sample combustion

.2.8. Just prior to the combustion analysis, a sample aliquot (20 - 100 mg) is weighed into a combustion boat and wetted with 1:1 HNO_3. The boat is then placed in the 70°C oven and excess HNO_3 driven off (not more than 15 minutes).

.2.9. Insert a combustion boat containing treated sample into the wire loop. Close the lid, and allow the instrument baseline to stabilize. This normally takes about two minutes.

.2.10. Press the "start" button, and slide the boat smoothly into the furnace at about two inches per second.

.2.11. After the instrument indicates the analysis cycle is complete, record the sample weight ignited and sample identification on the instrument printout.

.2.12. The same procedure is followed for blanks, samples, SRMs, and matrix spike samples. The size of the platinum boat and the percent carbon in the samples are the limiting factors for optimal sample size. Ten to 100 milligrams of prepared samples are used for analysis, with 40 mg preferred.

.2.13. Total analysis time is approximately 5-6 minutes.

.2.14. In some cases it may be necessary to analyze samples in quadruplicate. Check the SAM comments to determine whether this is necessary.

. Quantification

.1. µg sample burned

$$\mu g \text{ of sample burned} = \frac{W_s * 100}{(W_s + W_d) * TS_{105}} * W_b * 1000$$

ere:
W_s = weight (gm) sample diluted with sand
W_d = weight (gm) diluent sand used in sample preparation
W_b = weight (mg) sample combusted
TS_{105} = total solids (previously discussed)

.2. mg sand burned

$$\text{mg sand burned} = \frac{W_d}{W_s + W_d} * W_b$$

.3. "sand correction"

$$C_S = \text{mg sand burned } (W_b) * \text{Corr}$$

ere:
C_S = sand correction (response units)

.4. Net sample reading

$$R_S = R - C_S$$

ere:
R_S = net sample reading (response units)
R = uncorrected sample reading (response units)

.5. TOC result

$$\%\text{TOC} = \frac{R_s * CF * 100}{\mu g \text{ sample burned}}$$

Reports

. Sample Results and Quality Control Reports

.1. In addition to the sample results, all results for quality control tests are entered into the lab data base using the QC_DB program. These results include those for the method blank analyses, matrix spike results, duplicate results, and Standard Reference material (SRM) reports. It is not necessary to include a copy of the information entered into the QC_DB program with each job. However, a copy of the QC_DB Report form MUST be included in order to indicate that the QC was evaluated for analytical consistency.

.2. Optional reports which may be provided on client request include initial and continuing calibration information and raw data.

.. Control Chart(s)

.1. The recovery values for the LCS/SRM are plotted on control charts which may also be supplied to clients upon request.

.3. Definitions:

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

. **References**

.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA, 1986

Appendix I

QC Summary Table

Laucks Testing Laboratories

Method SW 9060 (modified for soil/sediment) QA Requirements and Corrective Actions

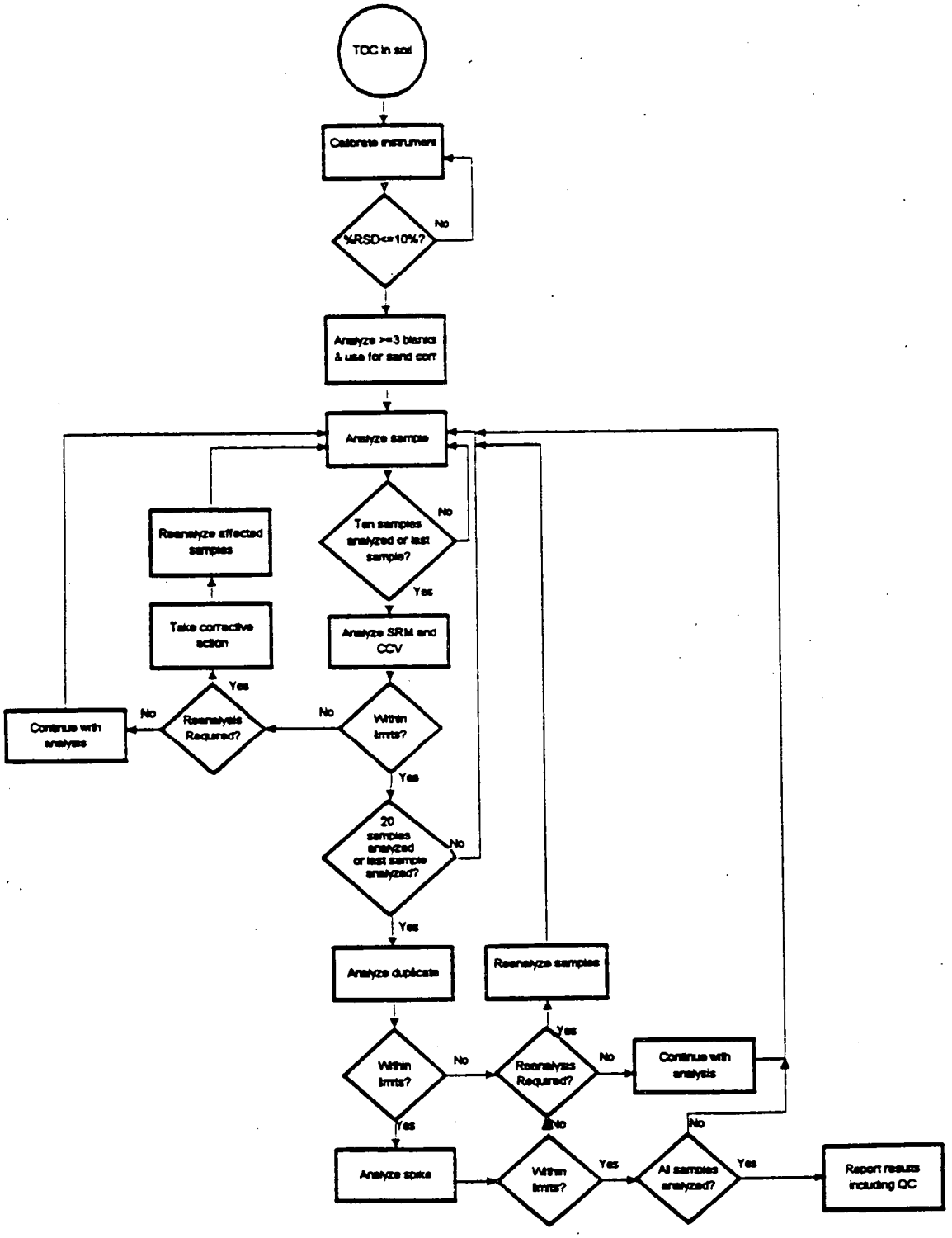
QA Element	Method Criterion (for water)	Laucks Criterion	Frequency	Corrective Action	Documentation
Initial Calibration	per manufacturers instructions	4-point curve 10 % RSD of calibration factors	with each analytical run	Fix problem and re-calibrate	in raw data package
Initial Calibration Verification	verify with an independently prepared check standard	verify with 1000 mg/L glucose solution	once, immediately following initial calibration	make up new ICV and/or calibration solutions; service instrument; report no analyses from failed ICV	in raw data package
Continuing Calibration Verification	verify with an independently prepared check standard every 15 samples	verify with a mid-point standard; CCV must fall within 10% of the true value	after every 10 samples	Fix problem & recalibrate; re-analyze samples which are not bracketed by in-control CCVs	in raw data package
Reporting Limit standard recovery	N/A	Must meet initial calibration criteria	Reporting limit standard is incorporated into curve	Recalibrate	in raw data package
Instrument Blank	N/A	Must be ≤2 ppm instrument reading	Once after initial calibration	Check system for leaks; check sample introduction technique; do not analyze samples	in raw data package

Method Blank (sand blank)	Once per sample batch; no criteria	Diluent (sand) blank is determined and sample results are blank corrected	Once per batch in at least triplicate	None; If blank is very high, diluent sand should be re-ignited	in raw data package
Matrix Spike Recovery	once every 10 samples; no criteria	50% - 150% unless criteria are published in lab control limits catalog or QC database	once every 20 samples	Review data; Consult QC Officer; some conditions may require re-analysis	narrative comment in report and raw data package
Duplicate % Difference	N/A	20% unless criteria are published in lab control limits catalog or QC database	once every 20 samples	Review data; Consult QC Officer; some conditions may require re-analysis	narrative comment in report and raw data package
Standard Reference Material (SRM) Recovery	N/A	NIST Buffalo River Sediment 2704 unless replaced	once every 20 samples	Review data; Consult QC Officer; some conditions may require re-analysis	narrative comment in report and raw data package

Appendix II

Flow Chart

TOC Analysis of Soils
by modified SW 846 Method 9060



Appendix III

Total Organic Carbon TS_{105} Verification Form

Laucks Testing Laboratories

Total Organic Carbon TS_{105} Verification Form

al organic carbon in soils and sediments are analyzed from samples dried at 70°C but reported as TOC :d at 105°C. Laucks generally considers any difference in total solids between these two temperatures to ninimal and should have little or no effect on the reported TOC. However, in order to demonstrate this, ıe samples from each project are dried at 70°C and a total solids at 105°C is performed on the dried ıple. If the results of these values are >90% TS (<10% moisture between these two temperatures), no :e secondary total solids determinations are made and all TOC values from the samples dried at 70°C are orted uncorrected for any additional moisture. A copy of this form and with any pertinent data should be .ched to each TOC data file in order to explain and demonstrate that this has occurred.

ny of these samples are <90% TS, all samples in the project will be analyzed for TS_{105} and the results d to correct the determined TOC values.

ent: _____

ject: _____

DG	Workorder	Sample No.	Net sample, grams dried @ 70°C (W_{70})	tare weight of container (grams)	70°C Sample dried @ 105°C + tare (W_{105})	% Total Solids (TS_{105})

Appendix IV

Method Bench Notes

Laucks Testing Laboratories, Inc. 4/9/97
Addendum to Laucks SOP LTL-9116, Revision 1

We are proposing the following deviations from Laucks SOP LTL-9116 for the analysis of Total Organic Carbon in Soil Samples for the NIROP - Fridley, Minnesota Project

Quantitation:
All sample quantitations will be reported as mg/kg, dry basis (DB) instead of %. The sample results will be recorded on a bench sheet similar to the one attached.

Data Package:
The equivalent raw data information and benchsheets will be provided in the package demonstrating the reporting of the TOC values in mg/kg DB on both the hardcopy and EDD.

The above procedural changes will supersede the Laucks SOP LTL-9116 for the duration of the project.

Bill Lundberg — 4/9/97
Reviewed by Bill Lundberg, Laucks Inorganic Supervisor — Date

Harry Romberg — 4-9-97
Approved by Harry Romberg, Laucks QA Manager — Date

Daneen C. Resnick — 4/11/97
Approved by Daneen Resnick, Brown & Root — Date

Other Approval — Date

Laucks Testing Laboratories, INC.
total organic carbon - soil

T[illegible] Organic Carbon	Item	Date	Method	Analyst	Reviewer	Analysis
J[illegible]97	Prep:		9060M	RLA		KC_TOC
Matrix: SOIL	Analysis:		SW846			TOC_ES

Sample ID	Sample (g)	Sand (g)	Samp+sand burned (mg)	Read	sample burned (ug)	Sand burned (mg)	Sand corr.	Net Read	ug C	mg/kg C	
ICV	1.000	0.000	40.0	989	40000	0.0	0.00	989.0	38.16	954.04	
ICB	1.000	0.000	40.0	11.93	40000	0.0	0.00	11.9	0.46	11.51	
PBS1	1.000	0.000	36.0	32.32	36000	0.0	0.00	32.3	1.25	34.64	
SRM17095	0.500	9.500	25.1	873	1255	23.8	17.64	855.4	33.00	26299	26000.
001-01	0.200	4.800	20.0	750	800	19.2	14.20	735.8	28.39	35489	35000.

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-9128

Procedure for the Determination of the Oxidation-Reduction Potential in Water or

n history:

	Date
:	04/09/97

by: Bill Lundberg — Date: 4/9/97
Bill Lundberg

ed by: Harry Romberg — Date: 4-9-97
Harry Romberg, QA Officer

ed by: Karen J. Kotz — Date: 4/9/97
Karen Kotz, Laboratory Director

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Introduction and Scope

1.1 Method Description

1.1.1 This method covers the procedure for the electrometric measurement of oxidation-reduction potential (ORP) in water or soil.

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 Oxidation-Reduction potential is defined as the electromotive force between a noble metal electrode and a reference electrode when immersed in a solution.

Equipment List and Standards

2.1 Equipment

2.1.1 pH meter.

2.1.2 Reference electrode - a calomel, silver-silver chloride nonflowing junction type.

2.1.3 Oxidation-Reduction electrode - a silver electrode or equivalent noble metal electrode.

2.2 Reagents

2.2.1 Water: ASTM Type II

2.2.2 Aqua Regia- Mix 1 volume of reagent grade concentrated nitric acid with 3 volumes of reagent grade hydrochloric acid.

2.2.3 Nitric Acid, reagent grade (1 + 1) - Mix equal volumes of concentrated nitric acid and water.

2.2.4 Sulfuric Acid - reagent grade.

2.2.5 Calcium Chloride - prepare a 1:1 calcium chloride solution with deionized warter.

2.3 Standards

2.3.1 Phthalate Reference Buffer Solution (pH_s = 4.00 at 25 degrees C) - Dissolve 10.12 grams of potassium hydrogen phthalate in water and dilute to 1 liter.

2.3.2 Phosphate Reference Buffer Solution (pH_s = 6.86 at 25 degrees C) - Dissolve 3.39 grams of potassium dihydrogen phosphate and 3.53 grams of anhydrous disodium hydrogen phosphate in water and dilute to 1 liter.

2.3.3 Redox Standard Solution; Ferrous-Ferric Reference Solution - Dissolve 39.21 grams of ferrous ammonium sulfate, 48.22 grams of ferric ammonium sulfate and 56.2 ml of sulfuric acid in water and dilute to 1 liter. The solution should be stored in a closed

glass or plastic container. The Ferrous-Ferric Reference Solution is fairly stable with a measurable oxidation - reduction potential.

2.3.4 Redox Reference Quinhydrone Solutions - Mix 1 liter of pH 4 buffer solution (see 2.3.1) with 10 grams of quinhydrone. Mix 1 liter of pH 7 buffer solution (see 2.3.2) with 10 grams quinhydrone. Be sure that excess quinhydrone is used in each solution so that solid crystals are always present. These reference solutions are only stable for about 8 hours so they must be prepared fresh for each day of analysis. The following table lists the nominal millivolt redox readings:

Nominal ORP of Reference Quinhydrone Solutions						
	ORP = v mV					
uffer Solution- ominal pH	4			7		
emperature, °C	20	25	30	20	25	30
eference Electrode						
g/Ag Chloride	268	263	258	92	86	79
alomel	223	218	213	47	41	34
ydrogen	470	462	454	295	285	275

afety precautions and Waste Disposal

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 Caution must be taken when handling acids to prevent burns.

2 Waste Disposal

3.2.1 Waste segregation and disposal from the point of collection is further covered in the Laucks SOP on hazardous waste disposal.

alibration and Quality Control

4.1.1 Before using electrode type meters allow them to warm up thoroughly. Bring them to electrical balance by carefully following the manufacturer's instructions. Set the scale or range to the millivolt level expected in the test solution.

4.1.2 Verify the sensitivity of the electrodes by noting the change in millivolt reading when the pH of the test solution is altered. The ORP will increase when the pH of the test solution decreases and the ORP will decrease if the test solution pH is increased. Place the sample in the beaker and agitate the sample. Insert the electrodes and note the ORP or millivolt reading. Add a small amount of a dilute NaOH solution and note the value of the ORP. If the ORP drops sharply when the caustic is added, the electrodes are sensitive and operating properly. If the ORP increases sharply when the caustic is added, the polarity is reversed and must be corrected in accordance with the manufacturer's instructions. If the ORP does not respond as above when the caustic is added, the electrodes should be cleaned and the procedure repeated.

4.1.3 Duplicate Readings

4.1.3.1 Perform a minimum of two successive readings on different sample portions per section 5.1.5 and report both results.

Operation procedures

5.1.1 After the assembly has been checked for sensitivity wash the electrodes with three changes of water or by means of a flowing stream from a wash bottle.

5.1.2 Preparation for water samples:

5.1.2.1 Place the sample in a clean beaker or sample cup and insert the electrodes. Immediately proceed to 5.1.4.

5.1.3 Preparation or soil samples:

5.1.3.1 Place approximately 5 grams of homogenized soil in a clean beaker or sample cup. Add 1:1 Calcium Chloride/deionized water solution to the soil in sufficient quantity to make a slurry.

5.1.3.2 Gently stir the slurry for approximately 2 minutes. Immediately proceed to 5.1.4.

5.1.4 Provide adequate agitation throughout the measurement period. Read the millivolt potential of the solution allowing sufficient time for the system to stabilize.

5.1.5 Measure successive portions of the sample (repeating the sample preparation steps outlined above) until readings on two successive portions differ by no more than 10 mV.

5.1.6 Calculations:

5.1.6.1 If the meter is calibrated in millivolts, read the oxidation-reduction potential directly from the meter scale. This ORP is related to the reference electrode used in the measurement.

5.1.6.2 Calculate the oxidation-reduction potential of the sample, in millivolts, referred to the hydrogen scale as follows:

$$E_h = E_{obs} + E_{ref}$$

where:

E_h = Oxidation-reduction potential referred to the hydrogen scale, mV,

E_{obs} = Observed oxidation-reduction potential of the silver reference electrode, mV,

E_{ref} = Oxidation-reduction potential of the reference electrode as related to the hydrogen electrode, mV.

eports

Data Reporting

6.1.1 Report the ORP to the nearest 10 mV. Also report on the benchsheet the pH at the time of measurement.

6.1.2 All standard and reagent preparation must be documented in the Inorganics logbooks. All standards and reagents must be traceable to the original stock or neat material.

6.1.3 The analyst must record the following information on the analytical benchsheet: date, analyst initials, Laucks sample identification number, sample and quality control results.

6.1.4 Copies of the above documentation must be placed in each applicable workorder file for long term document storage.

eferences

7.1.1 Standard Practice for Oxidation-Reduction Potential of Water, ASTM D1498-76 (Reapproved 1981).

7.1.2 Phone conversation with Daneen Resnick (Brown & Root) from Rock Vitale, April 8, 1997 - modifications to the method to obtain soil ORP measurements.

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-9202

e: **Total Suspended Solids (Non-Filterable Residue) Using EPA Method 160.2 / Standard Methods 2540D and Total Volatile Suspended Solids Using Standard Methods 2540 E.**

ision history:

nber	Date
	7/11/94
	3/7/97

CONTROLLED

tten by: Harry Romberg Date: 3-7-97
Harry Romberg, Quality Assurance Officer

iewed by: Bill Lundberg Date: 3/7/97
Bill Lundberg, Inorganic Conventional Chemistry Manager

proved by: Karen J. Kotz Date: 3/7/97
Karen Kotz, Laboratory Director

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ntroduction and Scope

Method Description

1 Scope and Application

1.1 This method is applicable to drinking, surface, and saline waters, domestic and industrial tes. The practical range of the determination is 4 mg/l to 20,000 mg/l. The detection limit, ever, is generally reported as 2 mg/l unless a sample volume greater than 100 mls is used.

1.2 A well-mixed sample is filtered through a standard glass fiber filter. The filter and due are dried to constant weight at 103°C-105°C.

1.3 The filtrate from this method may be used for Total Dissolved Solids (Filterable idue).

1.4 If volatile suspended solids are to be determined, the filter and residue are then ignited at °C and the loss on ignition determined.

2 Interferences

2. amples high in dissolved solids (filterable residue), such as saline waters, brines and e wastes, may be subject to positive interferences due to soluble material which has not been quately washed from the filter. Care must be taken to ensure that an appropriate filtering aratus has been selected and the filter adequately washed in order to minimize this possibility.

2.2 Samples which are very high in suspended material or which have certain particle sizes y plug the filter causing difficulty in filtering. It may be necessary to reduce sample size in er to reduce this tendency.

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

Sample Collection, Sample Storage, Holding Times

.1.1 No preservation of the sample is necessary. Refrigeration or icing to 4°C, to minimize robiological decomposition of solids, is required. Analysis should begin as soon as possible l should in no case exceed 7 days from the date of collection. All exceptions, whether due to oratory or client cause, must be recorded on a Holding Time Violation Report (HTVR).

Definition of Terms

.1 This section defines terms and acronyms as they are used in this SOP. Other terms, such MS/MSD or method blank, are not defined here since it is assumed that the user of this SOP eady understands their more general meaning.

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

)L - Method detection limit - The lowest concentration a sample which will yield a positive result that is greater zero at a known level of confidence. MDLs are empirically determined by Laucks, although there is no known way to determine the MDL for the TSS or TVSS analyses.

M or LCS - Standard Reference Material or Laboratory Control Sample. This is a material of approximately the same matrix as the samples, containing a known and usually certified amount of target analyte and which is prepared and analyzed in the same manner as a typical sample. This sample is used to demonstrate that the analytical system is in control. It may be considered to be a blank spike for most inorganic analyses and is preferred over artificially spiking blank materials. This type of sample is rarely analyzed at Laucks for TSS and there is no known source of TVSS material.

quence - A set of samples analyzed in a chronologically continuous group.

tal Suspended Solids (TSS) (nonfilterable residue)- Those solids which are retained on a glass fiber filter of the appropriate retentive ability and dried to constant weight at 103-105C.

tal Volatile Suspended Solids (TVSS) - Suspended solids which are volatile (burn off) at a temperature of 550°C.

Equipment List

ass fiber filter discs, 4.7 cm without organic binder, Gelman A/E or equivalent. Laucks rrently uses Pro Weigh 47 mm glass fiber filters from Environmental Express which are signed for TSS analysis. They are pre-washed and pre-weighed and require no additional eparation.

embrane filter funnel with the capability of adequately supporting 4.7 cm. filters

ction flask

ing oven. set at 103°C-105°C.

ffle furnace set at 550°C ± 50°C

siccator charged with active silica gel desiccant

alytical balance, capable of weighing to 0.1 mg.

Safety precautions and Waste Disposal

Safety Precautions

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

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

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

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

.5 Routine precautions include an awareness of elevated temperatures of both the oven and samples which have recently been removed from the drying oven or especially the muffle nace. The temperatures involved in this analysis can cause severe burns if adequate care is not en.

Waste Disposal

.1 No waste should be generated from this procedure. Sample residues may be washed out d discarded down the sink.

Quality Control

Method Blanks

.1 Criteria:

.1.1 Method blanks are used to verify contamination free reagents and apparatus. They are :pared with every set of samples prepared at the same time or at least one blank every 20 nples which ever is more frequent. The method blank is prepared by pouring the same amount deionized water through a filter as one would a typical sample (generally 100 mls.). Any TSS ove the detection limit is reported. Method blank control limits are such that contamination uld not exceed twice the detection limit. If 100 ml samples are used, Laucks generally nsiders the detection limit to be 2 mg/L with method blank control limits of 4 mg/L.

.1.2 In making a determination of whether or not the analysis is in control, the analyst should ually be normalizing the blank to whatever volume was used for any sample. It is assumed t any blank is due to washings from the filter and apparatus and is actually independent of nple volume. In other words, the control limit for this analysis is actually 0.4 mg. If blank ntamination exceeds that value, the TSS (or TVSS) of samples must be ten times that weight corrective action should be taken.

.1.3 If TVSS is being determined, the filters are processed in the same manner only are also ated to 550°C. Otherwise, the detection limits, criteria, and corrective actions are the same as TSS.

.2 Corrective action

.2.1 Corrective action may necessitate re-preparation and re-analysis of the sample set. For ample if TSS or TVSS 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 nk, the level of contamination may be considered insignificant. In any case, if re-analysis is t being undertaken, the analyst must first discuss the issue with the Quality Control Officer. In cases where blank contamination exceeds the control limit, a narrative comment must be de which documents the corrective actions taken.

2 SRM or LCS

2.1 Criteria

2.1.1 Analysis of a reference material is not normally required for TSS analysis and no known terial is available for TVSS. An SRM/LCS analysis will generally be analyzed only if ecifically required for a project. If not otherwise specified in that contract, it would typically

nalyzed at a frequency of once per 20 samples. Vendor specified control limits would be i for any such material.

2 Corrective Action

2.1 Re-analysis of all associated samples may be required if this sample exceeds it's limits. QC Officer should be consulted for any other corrective actions and all instances of out-of-rol events and any actions taken must be documented in the QC narrative of the report.

Sample Duplicate

1 Criteria

east one duplicate sample per 10 samples is required. RPD values are calculated as follows:

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

re

S1 = measured concentration in the initial analysis

S[illegible] measured concentration in the duplicate analysis

1.1 The RPD control limits are detailed in the Quality Control Database (QC_DB) and will nge from time to time. For samples with values which are less than 5 times the detection it, the control limit is equal to 5 times the detection limit. For values greater than 5 times the ction limit, the control limit is a calculated percent RPD.

2 Corrective action

2.1 If a trend in out of control RPD values is observed, the methods used must be examined etermine the source of variance. Once this source is identified, the method must be changed hat samples can be analyzed with a predictable reproducibility. If integrity of reported ple values is in doubt, re-analysis of all associated samples may be called for. Corrective ons should be discussed with the Quality Control Officer.

Operation procedures

Sample Analysis

Analysis sequence

Method Blank
SRM or LCS (if required)
up to 20 samples plus duplicates

Analytical Operation

.1 Preparation of glass fiber filter disc if Environmental Express Pro Weigh filters are being
: Remove the filter from it's aluminum weighing dish and place the disc on the membrane apparatus with the wrinkled side up. While vacuum is applied, rinse the disc with a small unt of deionized water in order to seat the disk in the filter device.

.2 Preparation of glass fiber filter disc if pre-washed and pre-weighed filters are NOT being
: Place the glass fiber filter on the membrane filter apparatus (if 4.7 mm filters are being) or insert into bottom of a suitable Gooch crucible (if 2.4 cm filters are being used) with the kled surface up. While vacuum is applied, wash the disc with three successive 20 ml mes of distilled water. Remove all traces of water by continuing to apply vacuum after r has passed through. Remove filter from membrane filter apparatus (if 4.7 mm filter) or crucible and filter if Gooch crucible is used, and dry in an oven at 103°C-105°C for one hour 550°C ± 50°C if TVSS is to be determined. Remove to desiccator and store until needed. eat the drying cycle until a constant weight is obtained (weight loss is less than 0.5 mg). gh immediately before use. After weighing, handle the filter or crucible/filter with forceps or s only. As in the procedure for the pre-weighed filters above, wet the filter slightly before n order to properly seat it in the filtration apparatus.

.3 Assemble the filtering apparatus and begin suction, taking care to have wet the filters as ribed in either procedure above. Shake the sample vigorously and rapidly transfer 100 ml to unnel by means of a 100 ml graduated cylinder. If this volume takes longer than 5-10 utes to pass through the filter, sample volume must be reduced such that the filtration time not be exceeded. If that volume is less than or equal to 10 mls, it should be dispensed with a al Mohr (graduated glass) pipet which has a wide enough tip opening so as not to inhibit the ing of solid pieces of material.

2.4 Filter the sample through the glass fiber filter, rinse the graduate and filter with three essive 10 ml portions of deionized water, allowing the rinsate to pass completely through the r between washings, and continue to apply vacuum for about 3 minutes after filtration is plete to remove as much water as possible.

2.5 If the sample contains large pieces of material which make it difficult or impossible to eve a representative, homogeneous sample, it may be necessary to thoroughly mix the ple in a blender prior to filtration. If this is done, however, care should be taken to assure air bubbles aren't entrained in the measured sample volume to the extent that it could affect actual volume dispensed. In other words, let the bubbles and subsequent foaming subside re dispensing the sample.

2.6 Carefully remove the filter from the filtration device, taking care not to leave pieces of r on the support apparatus, and place it in the aluminum weighing dish appropriate to that r and sample.

2.7 Dry the filter for at least one hour at 103°C-105°C.

2.8 Remove the filter and aluminum dish from the oven and place in a desiccator to cool. samples MUST be cooled prior to weighing or the apparent weight will be affected.

2.9 Repeat the cycle of drying, desiccating, cooling and weighing until the filter/dish attain a stant weight, changing by no more than 0.5 mg.

2.10 Record the final weight for calculation of TSS.

2.11 If TVSS is to be determined, repeat steps through using a muffle furnace pre-heated to °C ± 50°C. Record the final weight for calculation of TVSS.

Quantification

1 Residue concentrations are calculated using the following equations:

$$TSS = \frac{(W_f - tare) \times 1000}{V_i}$$

$$TVSS = \frac{W_f - W_a \times 1000}{V_i}$$

re:

V_i = volume of sample used in mls
W_f = weight of dried residue & filter
W_a = weight of ignited residue (after ignition @ 550°C) & filter in mg
tare = tare weight (weight in mg of filter before filtration of sample)

2 TSS and TVSS are generally reported on a mg/L as received basis.

6. Reports

6.1 Data Packet Organization

6.1.1 The data package for this analysis consists of the data sheet and a quality control database (QC_DB) report form.

6.2 Quality Control Reports

6.2.1 All results for quality control tests are entered into the lab data base using the QC_DB program. Printouts of all data entered need not be included in the package. However, all must be referenced on the report form. This includes the blank and duplicate results and any other QC which might have been analyzed by special request (such as an SRM/LCS).

6.3 Sample Result Reports

6.3.1 Data Qualifying Flags

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

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

Appendix I

QC Summary Table

Laucks Testing Laboratories

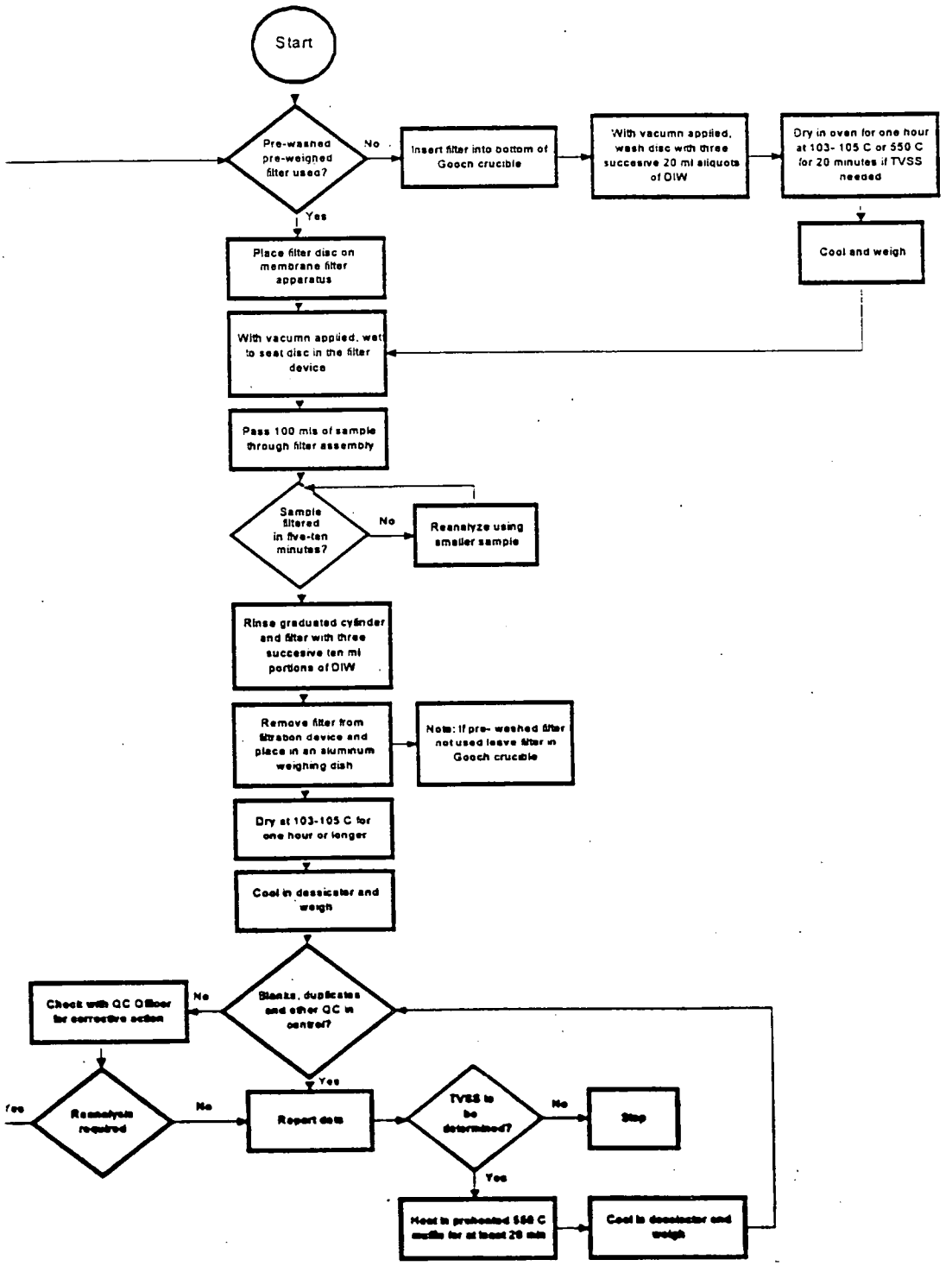
Method EPA 160.2 / SM 2540D & E QC Requirements and Corrective Actions

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Method Blank	None	<4 mg/L or no more than twice the detection limit	5% frequency (1 per 20 samples)	Re-analyze all samples <10x the actual weight of the solids. Consult QC Officer for any other actions	QC_DB report form with appropriate commentary
Duplicate % Difference	None	See current control limits catalog or QC_DB database	10% frequency (1 per 10 samples)	Discuss with QC Officer. If impact appears serious, may need to re-analyze samples	QC_DB report form with appropriate commentary
Standard Reference Material (SRM) Recovery	None	Within vendor supplied limits or 90%-110% recovery	If required, at frequency specified, or 5% if not specified	Discuss with QC Officer. If impact appears serious, may need to re-analyze samples	QC_DB report form with appropriate commentary

Appendix II

Flow Chart

Total Suspended Solids (TSS) and Total Volatile Suspended Solids (TVSS)
EPA 160.2 and/or SM 2540D&E



LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-9301

Title: **Spot Test for the Presence of Sulfide in Soil.**

Revision history:

Number	Date
0	04/09/97

Written by: Jim Owens, Principal 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 This method covers the procedure for the qualitative determination of sulfide in soil.

2. Equipment List and Standards

2.1 Equipment

2.1.1 Test tube, 10 to 20 ml.

2.1.2 150ml. to 250 ml. beaker

2.1.3 Filter paper, laboratory grade

2.1.4 Graduated cylinder, 5 or 25 ml.

2.1.5 Lead Acetate test paper - use commercially available lead acetate paper or prepare it in the following manner. Soak laboratory grade filter paper in saturated lead acetate solution until wetted. Remove from the solution and air dry.

2.2 Reagents

2.2.1 Hydrochloric acid - prepare a 20 - 30 % solution of hydrochloric acid in deionized water.

2.2.2 Saturated lead acetate solution - add approximately 20 grams of lead acetate to a beaker. Add 100 ml. of room temperature deionized water and stir.

3. Safety precautions

3.1 Safety Precautions

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

4. Quality Control

4.1 Laboratory Duplicate

4.1.1 At least one sample duplicate per 10 samples is required.

4.2 Corrective Action

4.2.1 The duplicate portion of the sample should reproduce the same qualitative results as the initial aliquot. If the duplicate results do not confirm the first analysis the sample should be mixed thoroughly and two new aliquots taken for confirmation

5. Operation procedures

5.1.1 Add approximately 1 - 2 grams of representative soil to a test tube or other appropriate container. If the soil is lumpy, gently break up the sample using a mortar and pestle, if necessary.

5.1.2 Moisten previously prepared or purchased lead acetate paper with a few drops of water.

5.1.3 Add approximately 5 mls. of the 20 - 30% Hydrochloric acid to the test tube and shake or mix for about ten seconds.

5.1.4 Immediately cover the test tube with the wetted lead acetate paper.

5.1.5 If sulfide is present the lead acetate paper will turn black within 3 to 5 minutes. The odor of Hydrogen sulfide may also be observed.

6. Data Reporting

6.1.1 All reagent preparation must be documented in the Inorganics logbooks. All reagents must be traceable to the original stock or neat material.

6.1.2 The analyst must record the following information on the analytical benchsheet: date, analyst initials, Laucks sample identification number, sample and quality control results.

6.1.3 Copies of the above documentation must be placed in each applicable workorder file for long term document storage.

7. References

7.1.1 Standard Methods for Chemical Analysis, Fifth edition, Volume One - The Elements, by Wilfred Scott, pp. 903 - 904.

ANALYTICAL METHOD AM18

ANALYSIS OF C_1-C_4 HYDROCARBONS IN WATER

1.0 Scope and Application

1.1 Method AM18 may be used to determine the concentration of dissolved gases in water samples. Specifically, Method AM18 may be used to determine the dissolved concentration of the following light hydrocarbon gases:

methane
ethane
ethylene
propane
propylene
i-butane
n-butane

1.2 This method is recommended for use by, or under the supervision of, analysts experienced in sample preparation, the operation of gas chromatographs and in the interpretation of chromatograms.

2.0 Summary of Method

2.1 Analysis of the C_1-C_4 hydrocarbons in a water sample is accomplished by transferring 30 ml of the sample plus 10cc of helium into a 50cc gas tight syringe. After equilibration, the headspace gases are analyzed with a gas chromatograph, using a backflush pre-column 10 port valve configuration and a flame ionization detector (FID). The sample (and standard calibration gas) is introduced into the columns by the mechanical injection of a sample loop. The data is transferred to a microcomputer where it is converted to digital format, stored, and processed using a chromatography data system (Chrom Perfect Direct, Justice Innovations).

3.0 Interferences

3.1 Ambient air is a potential source of "interference". Concentrations of methane in ambient air are typically 1.5 parts per million by volume (PPMV). Other light hydrocarbons may also be present at concentrations levels of concern. The analyst must take great care to ensure that air is flushed from the 50cc gas tight syringe before sample preparation and that no air has entered the syringe or needle prior to injection of the sample into the gas chromatograph.

3.2 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. An unrestricted flow of pure helium from a 10 psig source should be allowed to flow through the sample loop for 30 seconds prior to each analyses.

3.3 The analyst should demonstrate the absence of carryover contamination by analysis of the contents of the sample loop when purged with helium. This demonstration should be performed prior to the analysis of a sample set and when carryover contamination is suspected (after high samples). In the event that 'ghost peaks' (peaks similar to previous sample) appear when a pure helium sample is analyzed, measures should be taken to eliminate the carryover contamination.

3.4 Extra peaks in a chromatogram can be actual peaks from a previous run. Contamination from late eluting peaks can occur when the time between successive injections is too short.

3.5 The analyst should be certain that all peaks have eluted from the previous analysis prior to analyzing any sample or standard. If samples or standard chromatograms contain suspected 'extra peaks' the sample should again be analyzed after a clean baseline is established.

4.0 Apparatus and Materials

4.1 Sample vials: 40 ml VOA glass vials (QEC #2112-40ml or equivalent). Vials should be free of all hydrocarbons and compounds of interest prior to use.

4.2 Septa: Foil faced silicon (Integrated Liner Technologies, Inc.).

4.3 Syringe: Hamilton 50cc locking gas tight (#1050TLL or equivalent).

4.4 Gas Chromatograph: The chromatograph is equipped with the following: column oven, pre-column, analytical column, flame ionization detector, injection port, sample valve and sample loop. The column and detector for determination of C_1-C_4 hydrocarbons are a granular 3 ft. x 3/16 in. alumina analytical column and a flame ionization detector. The alumina column is protected against contamination by heavy organics by a 3 in. x 3/16 in. pre-column which is back-flushed after butanes have entered the analytical column. This arrangement allows rapid turn-around for consecutive analyses and a clean baseline for accurate, reproducible results. The flame ionization detector is of a special design which allows considerably more sensitivity than commercially available models.

4.5 Data Collection: The output of the chromatograph is directed to a microcomputer where the signal is converted to digital format, stored, and processed using a chromatography data system (Chrom Perfect Direct, Justice Innovations, Palo Alto, CA).

5.0 Sample Preparation and Analysis

5.1 Remove the sample (VOA) vials from the refrigerator. Let the samples reach ambient temperature over a period of 2 hours.

5.2 Using a clean 50ml gas tight, locking syringe withdraw 30ml of water from the bottom of the sample vial.

5.3 Withdraw 10cc of helium from a reservoir and lock the syringe.

5.4 Shake the syringe by hand (or use a wrist action shaker) for five minutes.

5.5 With the syringe in a vertical position, slowly inject the 10cc of headspace gas into the gas chromatograph sample loop though a septum fitting. The sample loop should be switched into the carrier gas flow stream (ten port valve activated) immediately after the sample loop has been filled with sample at atmospheric pressure. The flow through the sample loop is monitored by a flow meter connected to the sample loop vent port on the gas chromatograph.

6.0 Calibration and Results

6.1 The standard calibration gas should be introduced in the same manner as described in section 5.5 above. Measured peak areas are converted to concentrations in parts per million by volume using certified commercial gas standards traceable to NIST standards. (Matheson Gas Products Inc., or Scott Specialty Gases). Dilutes may be made to achieve multi point calibration curves.

6.2 At the beginning of a project or sample set, standards of appropriate calibration ranges will be run at least three times or until the results agree with a percent standard deviation no greater than 10%.

6.3 The instrument response (for any one subsequent standard in section 6.1 above) must not vary by more than 20%.

6.4 Concentration of analytes in the headspace gas in PPMV are converted to the original analyte concentration in the water (ng/l) using the following formula:

$$C_{WO}=\frac{(P)\,(V_G)\,(MW_X)\,(\frac{V_T}{V_G}-1+H_X)}{(R)\,(T)\,(H_X)\,(wt_W)}\times C_H\times 10^{1/2}$$

where:

C_{WO} = *original concentration of compound dissolved in water in nanogram per liter*
C_H = concentration of compound in headspace gas in parts per million by volume
MW_X = molecular weight of compound
H_X = distribution coefficient for compound X at room temperature
T = Room temperature (295.5 deg. K)
P = pressure = 1 atm
R = the gas constant = 82.07 cc atm / mole °K
V_G = volume of headspace gas
V_T = volume of liquid plus volume of headspace gas
wt_W = weight of the water

7.0 Quality Control

7.1 If the parameters set forth in section 6.3 are not met, the analytical program will be terminated until the cause is determined and a solution is effected.

7.2 The analyst should demonstrate the absence of ambient air and other contaminates in the sample preparation system by filling a sample syringe with helium and injecting 10cc of helium into the sample loop in the same manner as a sample. The results of this 'syringe blank' should demonstrate that C_1-C_4 hydrocarbon concentrations are below the minimum detection levels.

7.3 Before and during sample analysis, instrument blanks (sample loop filled with flush helium) should be analyzed to assure the absence of interferences as described in section 3.0 above.

7.4 Standards analyzed during the course of analyzing samples may be averaged into the calibration table as well as being used for peak identification. All chromatograms should be examined by an experienced analyst.

7.5 Throughout analysis the gas samples are injected mechanically utilizing a sample loop to achieve a uniform sample size from a flow

directly from the sample preparation syringe. The uniform sample size assures consistent and accurate results.

7.6 The water sample is withdrawn from the 40ml VOA vial through the septum using a 5 inch large bore luer lock needle while replacing the water with pure helium. The 30ml of sample is withdrawn from the bottom of the 40ml vial and the remaining sample is discarded.

7.7 Calibration records are generated and stored. All such records will be maintained in the laboratory during the course of the project and there after as determined by the client.

8.0 Instrument Conditions

8.1 Gas Chromatograph:

Injection Temp. ambient
Flame Ionization Detector Temp. ambient
Oven Temp. 100 deg. C. isothermal
Initial F.I.D. Signal Range 10E9
Carrier Gas Regulator 24 psig.
Hydrogen Pressure 22 psig.
Flame Air Pressure 25 psig. (1.0 scfh)

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

Written by: Harry Romberg
Harry Romberg, QC Officer

Date: 12-27-95

Approved by: Karen J. Kotz
Karen Kotz, Laboratory Director

Date: 12/28/95

UNCONTROLLED

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DOCUMENT CONTROL FORM 9

APPENDIX B 11

DOCUMENT CONTROL STAMP 11

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REPLACED VERSION STAMP 12

APPENDIX D 13

SOP UPDATE FORM 13

1. Introduction and Scope

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. 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 Manual or Chemical Hygiene Plan.

1.3. 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.

1.4. 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 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 must 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 is in order to make sure the responsible parties are all informed of the initiation of the creation or revision process. This form must 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:

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 8080."

2.2.4. The database, as a minimum, will track the document number, revision number, title and SOP Manual distribution. In addition, other information may be tracked where appropriate and might include responsible individual, current status (first draft, first review, final revision, final review, complete, etc.), and any other details that may appear necessary in order to facilitate completion of the document.

2.2.5. SOPs, Methods, and many other documents should have header information which clearly indicates the document number, revision, date of revision, document replaced by revision and, usually, page number. The header may vary from but should contain all appropriate information similar to the following:

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, QC Officer, and, where appropriate, Technical Director, Division Managers, etc.), it is added to the SOP 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 tracks the revision history of all documents. This database includes both current documents and their predecessors. Outdated documents and prior revisions are kept on file, with the intent of incorporating them directly into the database, but are generally not made available to analysts.

2.2.8. 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. These departmental copies are stamped in red with a Controlled Document Stamp (See Appendix B). 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 will be black on subsequent secondary copies and will not be directly tracked by the QA department as these documents are considered uncontrolled.

2.2.9. 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).

2.2.10. It is inappropriate for any individual to be working from an unapproved copy of a method or procedure.

2.2.11. It is inappropriate to make copies of the copies which are not stamped in red with the Controlled Document Stamp.

2.2.12. When documents are distributed to the departmental supervisor, a copy of the signature list(s) for the specific document(s) is 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. In addition, as staff new to a particular task (SOP) are trained, the departmental supervisor will ensure that they have read and signed the signature list for that SOP.
- The departmental supervisor is responsible for ensuring that all outdated versions of SOPs are discarded or destroyed.

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 copies 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 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.

2.3.6. When a document has been revised and the outdated version has been removed from circulation, the master copy of the outdated version will be stamped with the "Replaced Version" stamp (Appendix C) to ensure that it is never inadvertently used as a current version. In addition, replaced versions will be filed in the "history" file which is separate from the current versions.

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 D) must be used. A copy of this form is located in Appendix D. 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. SOPs should be reviewed approximately annually. Items addressed in the "SOP Update" forms will then be incorporated into the SOP itself. 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

Document Control Stamp

Controlled Document

Individuals must not perform work using this document unless they have signed the master and have been assigned a numbered copy. Unassigned copies will not be updated when changes occur.

No. _____

Assigned to: _____

Appendix C

Replaced Version Stamp

This document has been replaced by:

Revision: _____

on
Date: _____

Appendix D

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.

_____

_____

_____

_____

_____

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_____

Submitted by: _____ Date: _____

Approved by (QA): _____ Date: _____

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #~~LTL-COC~~ PK 12/26/95
LTL-1003

Title: Chain-of-Custody and Documentation Procedures

Revision history:

Number	Date
1.0	02/13/95

Written by: Harry Romberg, Quality Control Officer Date: 2-13-95

Reviewed by: Mike Nelson, Technical Director Date: 2-13-95

Approved by: John Buerger, Laboratory Director Date: 2/13/95

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LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP No. **LTL-1003**

Previous SOP No. **LTL-COC**

Title: **Chain-of-Custody and Documentation Procedures**

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.

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1. Introduction and Scope

1.1. Description

1.1.1. This SOP is intended to describe the chain-of-custody process at Laucks, for all samples from the point of receipt until the time of 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

2.1. Secure Storage Custody Log(s), see Appendix A

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, NEESA, 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 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 small refrigerator just outside of the walk-in is for volatiles sample storage.
- 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. For samples being logged into both the non-volatiles areas and volatiles refrigerator, a carbon copy of the Secure Storage Custody Log will be created for that refrigerator and the volatiles samples will subsequently be logged in and out using that form.

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.
- If they are being removed for transferal to another location (extractions or one of the volatiles storage locations), 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. Any analyst removing samples from **any** secure storage area for the purpose of preparation or analysis or transferral 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."

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. At least quarterly, the Secure Storage Custody Logs are collected by the Quality Control Department and collated into binders 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.

Appendix A

Secure Storage Custody Log

Laucks Testing Laboratories, Inc.

Secure Storage Custody Log

.L[illegible]nber: _____ Project: _____

›rage Unit: _____ SDG Number (*optional*): _____

mple Numbers	Matrix (*optional*)	Location (*shelf*)	Logged In Date	Logged In Time	By	Logged Out Date	Logged Out Time	By	Action

ıples Disposed of by _____ on _____

02/08/95

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1004

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

Revision history:

Number	Date
1.0	01/31/95
2.0	12/27/95
3	6/23/96

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

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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.*

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:	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, 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*				
Analytical Performance				
17 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 produce 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

GC Volatile Analyst Training Verification Checklist

nalyst Name:	Date:	Trainer:	Supervisor:	Analyst:
ocumentation				
Able to use Standards Log				
Able to use instrument Run Logs				
Able to use instrument Maintenance Logs				
ethods				
Has read and understands SOPs for all applicable methods				
List Method(s):				
Has read and understands EPA Methods				
List Method(s):				
Has read and understands appropriate sections of GC Training Manual				
strument Operation/Maintenance				
Knows location and use of Instrument Manuals				
Knows basic GC theory				
Able to use GC Control Pad to set temperature program				
Able to use Autosampler Control Pad to set injection program				
Able to check system flows				
Able to trim/change columns & perform leak check				
Able to measure and set carrier and makeup gas flows				
Able to bake column/injectors/detectors				
NON-ROUTINE: *Able to clean P&T and autosampler lines*				
NON-ROUTINE: *Able to change nickel tubing, resin and IPA*				
nalytical Performance				
Able to prepare standards & pass standard QC acceptance criteria				
Able to analyze and generate acceptable calibration curve				
Able to analyze CCVs and apply QC acceptance criteria				
Applies acceptance criteria for surrogates and spikes				
Able to set up analytical runs & acquire data				
Able to get information on samples/analyses (test codes, MDLs, etc.)				
Able to quantitate an analytical batch (standards, CCVs, QC & samples)				
Knows how to confirm detection of analytes (peak ID, conf. col.)				
Knows reanalysis criteria				
Able to perform sample dilutions (obtaining linear results)				
Knows correct reporting limits for method(s)				
Knows corrective action & documentation for out of control QC events				
Able to produce a data package (In-house and SW-846)				
ethod Validation *(complete one or more of the following)*				
Has successfully analyzed four P&A samples				
Has successfully analyzed two PE samples				
Has successfully analyzed three each of two types of QC samples				

is is to certify that _____ has been an analyst in the GC volatile department d has demonstrated competency at the preceeding tasks for the following methods (list below):

_____

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

Laucks Testing Labs

Fuels GC 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 & State Methods				
	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 & 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*				
Analytical Performance					
17	Able to prepare standards & pass standard QC acceptance criteria				
18	Able to analyze RTM standard and set up elution range				
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 & 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 reanalysis and reextraction criteria				
26	Able to perform sample dilutions (obtaining linear results)				
27	Knows correct reporting limits for method(s)				
28	Knows corrective action & documentation for out of control QC events				
29	Able to produce a data package (In-house and SW-846)				
Method Validation ***(complete one or more of the following)***					
30	Has successfully analyzed four P&A samples				
31	Has successfully analyzed two PE samples				
32	Has successfully analyzed three each of two types of QC samples				

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

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Laucks Testing Labs
HPLC Semivolatile Analyst Training Verification Checklist

nalyst Name:	Date:	Trainer:	Supervisor:	Analyst:
ocumentation				
Able to use Standards Log				
Able to use Instrument Run Logs				
Able to use Instrument Maintenance Logs				
lethods				
Has read and understands SOPs for all applicable methods				
List Method(s):				
Has read and understands EPA Methods (SW846)				
List Method(s):				
Has read and understands appropriate sections of HPLC Training Manual				
nstrument Operation/Maintenance				
Knows location and use of Instrument Manuals				
Knows basic HPLC theory				
Able to use solvent delivery system to set mobile phase program				
Able to use Autosampler Control Pad to set injection program				
Able to change filters and guard column				
Able to change columns & perform leak checks				
Able to measure and set mobile phase flows				
Able to prime pumps				
Able to prepare mobile phase (filter water, select correct solvent grade)				
Able to change Helium tank				
NON-ROUTINE: *Able to change lamps*				
NON-ROUTINE: *Able to locate the high pressure build-up*				
NON-ROUTINE: *Able to change pump seal*				
NON-ROUTINE: *Able to clean flow cell*				
nalytical Performance				
Able to prepare standards & pass standard QC acceptance criteria				
Able to analyze and generate acceptable calibration curve				
Able to analyze CCVs and apply QC acceptance criteria				
Applies acceptance criteria for surrogates and spikes				
Able to set up analytical runs & acquire data				
Able to get information on samples/analyses (test codes, MDLs, etc.)				
Able to quantitate an analytical batch (standards, CCVs, QC & samples)				
Knows how to confirm detection of analytes (peak ID, conf. col.)				
Knows reanalysis and reextraction criteria				
Able to perform sample dilutions (obtaining linear results)				
Knows correct reporting limits for method(s)				
Knows corrective action & documentation for out of control QC events				
Able to produce a data package (In-house and SW-846)				
ethod Validation *(complete one or more of the following)*				
Has successfully analyzed four P&A samples				
Has successfully analyzed two PE samples				
Has successfully analyzed three each of two types of QC samples				

is is to certify that _____ has been an analyst in the HPLC semivolatile partment and has demonstrated competency at the preceeding tasks for the following methods (list below):

_____

ms found to be not satisfactory at the 3 month interval should be discussed with the analyst and further ining 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.)			
15	Able to generate simple TIC data			
16	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:
	Able to Log-on to the RTE system	Observation
	Able to create and edit BLISTS	Successfully create 4 BLISTs
	Able to create spectra and quant reports	Done for 4 jobs
	Able to prepare sample extracts for analysis (including dilutions)	Done for 4 jobs
	Able to do basic mass-spec tuning	Submit 4 tune checks / observation
	Able to perform daily maintenance tasks	Observation
	Able to change a helium tank	Observation
	Able to enter data into SAM special tests and QC reports	Done for 4 jobs
	Able to get a basic directory listing of files on the RTE	Observation
	Able to use QAREA	Observation
1	Able to check a CCV standard for compliance to the method	Observation
2	Able to check DFTPP for compliance to the method	Submit 4 tune checks
3	Able to use basic RTE EDIT commands (create and edit files)	Observation
4	Able to use basic RPN commands (EC, DR, PF, PBM, etc.)	Pass RTE quiz at 85%
5	Able to generate simple TIC data	Done for 4 jobs
6	Able to check spectra vs. standard spectra	Done for 4 jobs
7	Applies acceptance criteria for surrogates, spikes, & ISs	Done for 4 jobs
8	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
0	Knows where to get information on samples (test codes, etc.)	Observation
1	Able to calculate RFs and results from raw data	Correctly complete 4 examples
2	Knows the types of extraction procedures used for ABNs	Observation
3	Knows basic GC/MS theory	Has read training manual
4	Has read and understands SOPs for all methods	Observation
5	Knows corrective action for out of control QC event	Observation / has read SOPs
6		
7		
8		
9		

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

Revised by: Harry Romberg
Harry Romberg, QC Officer

Date: 6-22-96

Approved by: Karen J. Kotz
Karen Kotz, Laboratory Director

Date: 6/22/96

UNCONTROLLED

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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 and Waste Disposal

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 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.

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

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

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

4.3.1 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.

4.3.2 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 contact 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 checks the zero on the balance. If it is off the user will adjust it according to the manufacturer's manual.

4.4.4 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.

4.4.5 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 the entry (See Appendix I). The standard weight will be a class "S" weight or equivalent.

4.4.6 The daily weight, after taring, must not vary from its nominal 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

4.4.6.1 **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.

4.4.6.2 **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.

4.4.7 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.

4.4.8 An example logbook page is presented in Appendix I

4.5 Weekly Calibration Check

4.5.1 The balance custodian is the person responsible to perform the weekly calibration check and to report 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 re 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.

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

APPENDIX I

Sample Page from a Balance Logbook

Class S

Week of:	Weight	Reading	Mon.	Tue.	Wed.	Thur.	Fri
June 16 - June 22, 1996	1.00	1.00	Inlet = 130	128.6	132.6	146.7	13
Harry Rombing	10.00	10.00	2gm = 2.00	2gm = 2.00	2gm = 1.99	2gm = 2.07	2gm =
	50.00	50.00					
6/17/96	150.00	150.01					
June 23 - June 29, 1996							

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP No. **LTL-1006**

Previous SOP No. **LTL-0053**

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

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #: LTL-~~0053~~ 1006 12/24/95

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

Submitted by: Vicki Talksabout Date: 10/13/93
Vicki Talksabout

Approved by: Harry Romberg Date: 10/13/93
Harry Romberg, QCO

Approved by: Mike Nelson Date: 9/27/93
Mike Nelson, Chief Chemist

Approved by: John M. Buerger Date: 10/11/93
John Buerger, Lab Director

UNCONTROLLED

OPERATORS STATEMENT

I have read the contents of this SOP and understand its use.

NAME	SIGNATURE	DATE

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1. **INTRODUCTION** - This SOP provides a description of initial calibration of thermometers used for refrigerators, freezers, and ovens and the system used to record the calibrations and locations of the thermometers.

2. **SAFETY** - 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. **MATERIALS AND SUPPLIES LIST**

Ertco Standard Thermometer- Stored in QC Officer's desk
High temperature grease pen
Disposable gloves
500 ml Erlenmeyer flask
Glass rod
Crushed ice

4. **IDENTIFICATION OF THERMOMETERS**

4.1. Refrigerator and Freezer Thermometers.

4.1.1. Thermometers are purchased from Streck Laboratories, Incorporated. Thermometers are received with an individual serial number imprinted on the thermometer and a certificate stating that the thermometer was calibrated in accordance with standards traceable to the National Institute of Standards and Technology. The Streck thermometers are immersed in a glass vial of forty-nine percent ethylene glycol. The entire thermometer and vial assembly is further encased in a plastic sleeve to prevent breakage.
Alternate thermometers may be purchased in the future if a thermometer superior to the Streck design should become available.

4.1.2. Certificates are logged in a binder labeled Certificates-Thermometers located in the QC department shelves.

4.2. Oven Thermometers.

4.2.1. Oven thermometers are purchased from commercially available sources such as VWR. At a minimum the thermometer should be a mercury filled thermometer and measure from zero degrees Celsius to 110 degrees Celsius. Most inorganic ovens require a thermometer capable of reading to at least 180 degrees Celsius.

4.2.2. Upon receipt, thermometers are marked with an ID number. Thermometers are marked using a special grease pen that will survive high temperatures. Currently, thermometers numbered one through five are used in inorganic ovens while six through eight are located in extractions.

4.2.3. If an oven thermometer is replaced, its successor will be the same number followed by a alpha sequence character. For example, the first replacement of thermometer 5 will receive the ID number of 5A.

5. CALIBRATION OF THERMOMETERS

5.1. Calibration of the standard thermometer.

5.1.1. The standard ERTCO thermometer is recalibrated yearly. The standard thermometer is currently recalibrated every September.

5.1.2. It is only necessary to perform the thermometer recalibration at one point. The ice point, 0 °Celcius, is considered a "fixed point" in liquid-in-glass thermometry and is therefore chosen as the recalibration point.

5.1.3. Before recalibration, the standard thermometer must remain at room temperature for seventy-two hours.

5.1.4. Preparation of the ice bath.

5.1.4.1. Materials needed will include: a 500 milliliter

Erlenmeyer flask, a glass rod, crushed ice sufficient to fill the Erlenmeyer flask, and a minimal amount of water.

5.1.4.2. Fill the flask with crushed ice and add minimal amount of water to create a slurry. Stir the ice water slurry with a glass rod and immerse the thermometer.

5.1.4.3. Put as much ice in the flask as possible.

5.1.4.4. Allow the bath to equilibrate for five minutes with occasional stirring.

5.1.4.5. When the temperature has remained stable for at least two minutes record the ice-point reading.

5.1.5. If the ice-point reading is found to be higher or lower then the previous calibration reading, all other readings will be higher or lower, respectively, by the same amount.

5.2. Calibration of refrigerator thermometers.

5.2.1. Streck (Temp-Chex) refrigerator thermometers are calibrated upon receipt and yearly thereafter. All currently used thermometers are recalibrated every July. When a thermometer has been recalibrated, a small color coded sticker is attached. The color code will correspond to a particular yearly calibration. For example, fall 1993 calibrations, correspond to a gold color sticker. Thus an analyst can easily know his/her thermometer is currently calibrated.

5.2.2. Thermometers are placed in the GC/MS locked volatiles refrigerator (R-04). This refrigerator was chosen due to the fact that it is not frequently opened.

5.2.3. At the same time the ERTCO standard thermometer is also placed in the volatiles refrigerator. The standard thermometer is placed in an Erlenmeyer flask of water.

5.2.4. The thermometers are allowed to equilibrate for forty-eight hours and the temperatures read and recorded. Read the temperature of the standard thermometer first, then the individual Streck thermometers.

5.2.5. The difference in the Streck thermometer reading from the standard thermometer reading and the actual Streck thermometer temperature reading is recorded to the nearest 0.1 °C into a logbook. Each thermometer has a page in the logbook identified by the thermometer's serial number. The logbook page lists the purchase date, the current location of the thermometer and the thermometers calibration history. This logbook was initiated in December 1992, so information prior to this date may not be contained in the logbook.

5.2.6. It is important to allow the thermometers to equilibrate for the full forty-eight hours if the thermometer has been stored at room temperature, or incorrect temperature deviation results may be obtained. If the thermometer has been in use at ordinary refrigerator temperatures, twenty-four hours is sufficient.

5.2.7. A minimum of one calibrated refrigerator thermometer shall be kept on hand as a spare.

5.3. Calibration of freezer thermometers.

5.3.1. Streck (Temp-Chex) freezer thermometers are calibrated upon receipt and yearly thereafter. All currently used thermometers are recalibrated every July. When a thermometer has been recalibrated, a small color coded sticker is attached. Thus an analyst can easily know his/her thermometer is currently calibrated.

5.3.2. Thermometers to be calibrated are placed in the GC/MS volatiles freezer (F-05). This freezer was chosen due to the fact that it is not frequently opened.

5.3.3. At the same time the ERTCO standard thermometer is also placed in the volatiles freezer. The thermometer being calibrated and the standard thermometer will need to be placed on the same freezer shelf.

5.3.4. The thermometers are allowed to equilibrate for forty-eight hours and the temperatures read to the nearest 1 °C and recorded. Read the temperature of the standard thermometer first, then the individual Streck thermometers. The standard thermometer should be measured first as the standard thermometer is not encased in liquid as the Streck thermometers are. This

will cause the standard thermometer's temperature to rapidly change when the freezer door is opened.

5.3.5. The difference in the Streck thermometer reading from the standard thermometer reading and the actual Streck thermometer temperature reading is recorded into the logbook page referenced by the serial number of the individual thermometer.

5.3.6. It is important to allow the thermometers to equilibrate for the full forty-eight hours if the thermometer has been stored at room temperature, or incorrect temperature deviation results may be obtained. If the thermometer has been in use at ordinary freezer temperatures, twenty-four hours is sufficient.

5.3.7. A minimum of one calibrated freezer thermometer shall be kept on hand as a spare.

5.4. Calibration of oven thermometers.

5.4.1. Oven thermometers are calibrated upon receipt and yearly thereafter. All currently used thermometers are recalibrated every August. When a thermometer has been recalibrated, a small color coded sticker is attached. Thus an analyst can easily know his/her thermometer is currently calibrated.

5.4.2. Thermometers to be calibrated are placed in a boiling water bath.

5.4.3. At the same time the ERTCO standard thermometer is also placed in the boiling water bath. The thermometers will read a temperature slightly above 100 degrees Celsius if the bulbs of the thermometers are resting directly on the bottom of the beaker while the hotplate is in a heating mode.

5.4.4. The thermometers are allowed to equilibrate for four-five minutes and the temperatures read to the nearest 1 °C and recorded in the thermometer logbook. The difference in the thermometer reading from the standard thermometer reading and the actual thermometer temperature reading is recorded into the logbook page referenced by the number of the individual thermometer.

6. RECORDING AND STORING OF DATA- Data for refrigerator/freezers and ovens is recorded into a logbook.

6.1. Refrigerator and freezer thermometer logbook.

6.1.1. Each thermometer has a page in the logbook identified by the thermometer's serial number. The logbook page lists the purchase date, the current location of the thermometer and the thermometers calibration history. This logbook was initiated in December 1992, so information prior to this date may not be contained in the logbook.

6.1.2. The thermometer calibration history includes the actual temperature recording of the thermometer being calibrated, the deviation in degrees from the standard thermometer reading, the date, and the initials of the person performing the calibration.

6.2. Oven Thermometer Logbook

6.2.1. Each oven thermometer has a page in the logbook identified by the thermometer's number. The logbook page lists the purchase date, a description of the type of the thermometer, the current location of the thermometer and the thermometers calibration history. This logbook was initiated in December 1992, so information prior to this date may not be contained in the logbook.

6.2.2. The thermometer calibration history includes the actual temperature recording of the thermometer being calibrated, the deviation in degrees from the standard thermometer reading, the date, and the initials of the person performing the calibration.

6.3. Standard Thermometer Logbook

6.3.1. The standard Ertco thermometer has a page in the logbook. The logbook page lists the purchase date, a description of the type of the thermometer, the current location of the thermometer and the thermometers

calibration history.

7. SPECIFICATION LIMITS AND CORRECTIVE ACTIONS

7.1. Specification Limits.

7.1.1. Princo Instruments Inc. specifies a +/- 1 ° Celsius specification limit on the Streck refrigerator thermometers. (Princo Instruments is the actual manufacturer of the thermometers.)

7.2. Corrective Actions.

7.2.1. If there is any visible break in the mercury, this must be corrected before calibration. Currently Clyde Ambacher in inorganics is handling repair of thermometers with mercury breaks.

7.2.2. Every three months a cold storage audit is performed. As part of this audit, all thermometers are checked for mercury breaks and other deterioration. If mercury breaks are discovered the thermometer is replaced with the calibrated spare and the faulty thermometer is repaired and recalibrated or replaced.

7.2.3. If a refrigerator or oven thermometer deviates from the standard thermometer by more than +/- 2 ° Celsius it should be repaired or replaced. If this is a new thermometer, the possibility of a mercury break should be doublechecked. If no mercury break or other easily discernible cause can be found, the thermometer should be returned to the manufacturer for replacement. If this is a used thermometer, undergoing yearly recalibration, the thermometer should be replaced.

7.2.4. If a freezer thermometer deviates from the standard thermometer by more than +/- 5 °Celsius it should be repaired or replaced. If this is a new thermometer, the possibility of a mercury break should be doublechecked. If no mercury break or other easily discernible cause can be found, the thermometer should be returned to the manufacturer for replacement. If this is a used thermometer, undergoing yearly recalibration, the thermometer should be replaced.

7.2.5 If the standard thermometer ice point deviates from 0.0 ° Celsius all temperature readings taken with the standard thermometer will have to be adjusted accordingly.

8. REFERENCES

8.1

U.S. Department of Commerce
National Institute of Standards and Technology
NIST Special Publication 819
August 1991
A Procedure for the Effective Recalibration of Liquid-in-Glass Thermometers

Appendix I- Sample Logbook Page

Thermometer Serial #: B 801708

Purchased: Streck/Temp Check prior to Aug 1992

Current Location: R-06 - Extractions - Brown Fridge

Calibration History

Actual Temp Recording	Deviation from Std Thermometer	Date	Initials
	−0.8°C	Aug 1992	VT
3.8°C	−0.4°C	Sept 2, 1993	VT

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP No. **LTL-1007**

Previous SOP No. **LTL-0045**

Title: **Maintaining Instrument Records and Logbooks**

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.

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LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-~~0045~~ 1007 12/26/95

Title: Maintaining Instrument Records and Logbooks

Submitted by: Harry Romberg Date: 9-9-91

Reviewed by: Mike Nelson Date: 9/9/91

Approved by: Jim Owens Date: 9/9/91

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.

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SECTION I - Introduction and Scope

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.

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.

SECTION II - Equipment List

1) maintenance logbook

2) analytical run logbook (where appropriate)

3) pen (pencil is NOT allowed)

SECTION III - Safety precautions

No safety related precautions need be observed in the performance of this SOP. However, special precautions are needed for the instruments and related chemistries. These precautions should be outlined in the respective operational SOPs.

SECTION IV - Operation procedures

1.0 General

1.1 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.

2.0 Maintenance Manuals

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.

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.

2.2.1 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.

2.3 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.

2.3.1 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.

2.3.1.1 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.

2.3.2 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.

2.3.3 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.

2.3.4 Each entry should be **INITIALED** by the person making the entry.

3.0 Instrument Run-Logs

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.

3.1.1 GC, GC/MS, HPLC, and GPC 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 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.

3.1.1.1 They 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 these logbook forms are located in Appendices I (GC/MS), II (GC and HPLC), and III (GPC).

3.1.1.2 In addition to the appropriate header information for each analytical GC, GC/MS, HPLC or GPC run, all of the pertinent information should be filled out for each injection.

3.1.1.3 The samples, standards, calibration checks, reference materials, etc. should be listed IN ORDER.

3.1.1.4 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).

3.1.1.5 No empty space should be left between daily logbook entries. The end of the analytical sequence should be clearly marked, the most common acceptable method is to mark the space in the logbook after the last entry with a /E. If both of these criteria are met, it is not necessary to cross out unused space at the end of the analytical run. This space may be used for subsequent notes AS LONG AS the end of the sequence is clearly defined.

3.1.2 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.

3.1.2.1 A copy of the run-log is included with each data packet associated with that run.

3.1.2.2 As with the bound book format, the samples, standards, calibration checks, reference materials, etc. should be identified and listed IN ORDER.

3.1.2.3 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.

3.1.2.4 It is not a current labwide laboratory practice to maintain ongoing run-logs for inorganic instrumentation, although individual instrument analysts may choose to keep this information available either by archiving computerized information on diskette or by keeping hardcopy versions. As the daily run-logs are included with all data, it is not currently considered necessary. It is advisable, however, to maintain such records in a bound (3-ring binder is OK), organized format and not unbound, loose-leaf.

APPENDIX I

GC/MS Run Log

LAUCKS TESTING LABORATORIES

GC/MS OPERATIONS LOG

ABN

ABN

Page

Case #

Run #	File Name	Sample #	Dilution		IS A RT	IS A Response	IS B RT	IS B Response	IS C RT	IS C Response
1	AIT7201									
2	HH09A	SSTD050	MS2-70-2	ORD 25						
3	MT7201									
4	HH09B	SSTD050	MS2-70-2	ORD25						
5	MT7201									
6	HH09C	SSTD050	MS2-70-2	ORD 25	~~97~~ 12.47	77061	16.28	181641	21.82	89817
7	HH09D	SSTD050	MS2-61-1	ORDDEG 20.	12.48	113027	16.29	270441	21.83	131253
8	HH090 HC-SFA	SBLKH1	B0202451BHI	16→2	12.49	71464	16.28	764818	21.83	79018
9	HH091	COMP1	9107B20-1		12.47	66036	16.28	1148807	21.82	72983
10	HH092	COMP1MS	-1MS		12.48	66671	16.27	151500	21.81	75210
11	~~HH093~~ HH093	MSD	-1MSD		12.55	66354	16.36	160601	21.90	84808
12	HH094	COMP2	-2		12.47	62835	16.28	151014	21.82	73633
13	HH095	COMP3	-3		12.49	65236	16.30	153226	21.84	75655

LAUCKS TESTING LABORATORIES

GC/MS OPERATIONS LOG

Page ~~3125~~ 2957

Case #

ABN: 1

Run #	IS D RT	IS D Response	IS E RT	IS E Response	IS F RT	IS F Response	Data Reduction	Comments, Changes	Anal
1								100 - 33.5 - 33.2 - 1.47	BP
2								DFTPP FAIL 441 ↓	
3								100 - 33.3 - 31.0 - 1.29	
4								DFTPP FAIL 441 ↓	
5								100 - 34.1 - 31.8 - 1.04	
6	26.45	134240	34.86	68027	—	—	HALEORD	OK.	
7	26.45	226661	34.87	107386	—	—	CHLORIDE	*NOTE USES IS 6 & 10 ng/ml	
8	26.45	124451	34.87	69954	—	—	ORDINUE & CHLORIDE		
9	26.47	123824	34.87	81690	—	—		Needs 1:20 for TNT	
10	26.47	121864	34.87	72707	—	—			
11	26.55	146291	34.95	96307	—	—			
12	26.49	134842	34.86	96254	—	—		Needs 1:20 for TNT	
13	26.49	137842	34.88	90919	—	—		Needs 1:20 for TNT	

APPENDIX II

GC/HPLC Run Log

GC INSTRUMENT LOGSHEET

OPERATOR DSS DATE 8-9-91 Case/lab number _____

CHROMATOGRAPHY REFERENCE PAGE _____

COLUMN USED _____ OVEN TEMP _____ deg C ISOTHERMAL

PACKED COLUMN FLOWRATE _____ ml/min FSCC LINEAR VELOCITY _____ cm/sec at ____ DEG

CALIBRATION STD REFERENCE SE0809

Seq #	Bot #	Run number	SAMPLE IDENTIFICATION	Dil	Ext vol	reference peak R.T.	Resp.	Comments
1	2	*RAW003	HEXANE					
2	4	RE1302	PX2-48-3					
3	6	RE1303	PX2-48-2					
4	9	RE1304	PX2-48-1					
5	10	RE1305	EX4-37-2/5E0					
6	12	RE1306	R0807GHBLOWA					
7	14	RE1307	R0807GHBLOWMA					
8	16	RE1308	#1 WELL GSR					
9	18	RE1309	#1 WELL MS					
10	20	RE1310	#1 WELL MD					
11	22	RE1311	PX2-48-1					
12	24	RE1312	ARCO 1000					
13	26	RE1313	ARCO 1000 MS					
14	28	RE1314	ARCO 1000 MD					
15	30	RE1315	PX2-48-1					
16	32	/E						
17	34							
18	36							
19	38							
20	40							
21	42							
22	44							
23	46							
24	48							
25	50							
26	52							
27	54							
28	56							
29	58							
30	60							

APPENDIX III

GPC Run Log

Reserved

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1008

Title: **QC Corrective Action**

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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 <u>reporting</u> 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: _____
QA Department: _____ Date: _____

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

Appendix 4

Sample Receipt Form

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

P:\PM\Holly\docs\fax\ncmemo.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:

Appendix 5

Example Logsheet (Cold Storage)

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												
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26												
27												
28												
29												
30												
31												

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

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 actions MUST be recorded on this log sheet. If there is insufficient room, mark on the back of the page with the date the action occurred.

Samples MUST NOT be stored in units which are not maintaining the proper temperature.

REFERFRM.DOC 5/29/96

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1009

Title: **Blind Spike Program**

Revision history:

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

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

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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**

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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) Inorganics Statement of Work (SOW).

1.1.3 Criteria for Precision and Accuracy (P&A) Studies are generally defined in the specific 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. MDLs are determined on each instrument used for organic analysis.

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. 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.

1.3 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.

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.

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 be assigned by QA.

6.2 Instrumental Detection Limits (IDLs)

6.2.1 It is not 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 MDL.

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.

6.2.9 It is strictly prohibited to compute MDLs based on IDL determinations.

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).

- Laboratory ID number
- Analyst who performed the preparation
- Method number of the preparation (where applicable)
- Date(s) the study was prepared
- 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 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, possibly 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

6.3.6 In order to determine the Method Detection Limit (MDL), it is first necessary to estimate what the MDL will be in order that the appropriate spiking levels may be used. How this estimate is made is immaterial to the actual MDL determination. Methods for making this determination may include any one or a combination of the following:

- estimating based on the instrument detection limit (IDL) as determined above or by any other means
- estimating based on the previous MDL
- estimating based on 3 times the instrument signal to noise ratio
- estimating based on analyst judgment

6.3.7 A solution is then prepared and spiked into a sample matrix, which is as free as possible of interference and target analytes, at a level that will result in a sample concentration equivalent to 1 to 5 times the estimated MDL.

Note: 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.

6.3.7.1 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.

6.3.7.2 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.

6.3.8 Preparation of Spiked Samples

6.3.8.1 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.

6.3.8.2 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.

6.3.9 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.

6.3.9.1 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

6.3.9.2 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, 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.

6.3.9.3 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

6.3.9.4 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.

6.3.10 Methodology Exceptions/Specifics

6.3.10.1 Wet Chemistry

6.3.10.2 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.

6.3.10.2.1 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.

6.3.10.3 GC and Gas Chromatography/Mass Spectophotometry

6.3.10.3.1 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.

6.3.10.3.2 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, possibly 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.

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, 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), ILM03.0.

EPA "600" Series Methods, section 8.1.1, 40 CFR, Part 136, App. A.

EPA SW846 "8000" Series Methods, section 8.6 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

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-~~0033~~ ~~12/24/95~~
1012

Title: Solvent QC Monitoring for Trace Residue Analysis

Submitted by: Harry Romberg — Date: 4-10-91

Reviewed by: Vicki Talksabout — Date: 4-11-91

Reviewed by: Mike Nelson — Date: 4/11/91

Approved by: Jim Owens — Date: 4/15/91

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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.

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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 containing 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, MECLAC. If the solvent fails, residue analysis SHOULD NOT be performed until a suitable acceptable lot is determined. The Extractions supervisor should see that any such failing lot has been terminated in SAM. Data and the report, however, should still be submitted to the 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

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LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1013

Title: **Preparation, Storage, Shelf Life and Traceability Documentation of Standards and Reference Materials**

Revision history:

Number	Date
1	8/31/92
2	4/17/96
3	6/3/96

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

Reviewed by: Monica Carr, Organics Manager — Date: 6/6/96

Reviewed by: Bill Lundberg, Inorganics Manager — Date: 6/6/96

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

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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

LAUCKS TESTING LABORATORIES

NEATS DATABASE PROGRAM

SEARCH BY ONE OF THE FOLLOWING PARAMETERS:

CAS Number | Bottle Number

Chemical | Synonym #1 | Synonym #2

Enter New Chemical

Open Report for Expiring Chemicals

Appendix 3

Neats Database Screens

Appendix 4

Logbook Examples

NEATS DATA ENTRY

Chemical	n-nitroso-diphenylamine	CAS #	86-30-6
Synonym #1		Purity (%)	98.3
Synonym #2		Lot #	153-1508
Bottle #	N167	Source	ChemService
Old Bottle #		Catalog #	O-374
Box #	2	Size (g)	2
Received	4/3/96		
Expires	10/1/2000		
Notes	For GC/MS SVOA MS/MSD		

Previous | Next | New | Exit

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)
HM4-03-04 con't 4	2,4,6 - TNT	M-8330-11	1000 ppm	10 ul	50 ml	200 ppb	DI H2O	12/30/95	MH	04/01/96	
⊥	HMX	M-8330-4	⊥	⊥	⊥	⊥	⊥	⊥	⊥	⊥	
01	1,3,5 - TNB	M-8330-12	1000 ppm	10 ul	50 ml	200 ppb	Fluid TCLP #1	12/30/95	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	⊥	⊥	⊥	⊥	⊥	⊥	⊥	⊥	

MA-1-1

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	KP 4/2/96 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	1000 ppm Zn								
2	ABN SPE ICP/MS ICV3	ME4-30-01	ICP/MS ICV-1	500 ul	5 mL		10% HNO3 H2O	4/3/96	ABN		
3	As	ME3-81-9 Exp 5/96	1000ppm	100 µl	5-11-96 100 mL	1ppm	5% HNO3	4-3-96	tm	5-96	
4	ICP/MS STD1	ME4-30-02	ICP/MS	50 µl	5mL		5% HCL H2O	4/3/96 ABN	ABN		
5	STD2		Starch Std	250							
6	STD3			500							

ME4-48

Appendix 5

Bench Sheet 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	IC ICV Cl	ERA 3412	—	—	250 ml	129 mg/L	—	logged in 3/18/96	SK	Opened: 4/5/96 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 µ	500 µl	100 ml	5 µ	H_2O	3/20/96	SK	4/3/96	
	Cl	.5	1000 µ	1000 µl		10 µ					
	NO_2	.7	100 µ	2000 µl		2 µ					
	NO_3	.3	1000 µ	500 µl		5 µ ~~10 µ~~ 4/2/96					
	SO_4	.6	1000 µ	2000 µl		20 µ					
3	NO_2 ICV	IC4.78.7	100 µ	1000 µl	100 ml	1 µ	H_2O	3/20/96	SK	3/27/96	
4	NO_2 ICV	IC4.78.7	100 µ	1000 µl	100 ml	1 µ	H_2O	3/25/96	SK	4/1/96	

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.% nonyl phenol	11.22	59100	14.98	168403	20.42	100469
4	HC180	Kalama	B0313961SVWLA	9603368-BLK	1000→1	11.23	58139	14.98	166207	20.43	99304
5	HC181	Kalama	S0313961SVWLA	—SPK	1000→1	11.24	55723	14.99	155289	20.44	98643
6	HC182	Olympia	—	9603266-01DL	1:4 1000→1	11.24	52364	14.98	158592	20.44	102071
7	HC183	Kalama	—	9603368-01	1:2 100→1	11.24	58997	14.98	168568	20.46	103091
8	HC184	↓	—	-01DL	1:4 100→1	11.24	58386	14.98	173480	20.45	106213

AB 3/19/96

As, Tl

toSampler Report Table: SW846 Wed 04-10-96 09:39:45 AM page 1

ble Name: SW846 Autosampler Type: TYPE TJA
mple Positions: 257/300 QC Positions: 11/19 # Sets: 1
nse Station location is rack -1, pos. -1.

- Racks ---

ck #	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 ---

t#	Type	Prepare?	Description	Method	#Pos	Rack#	StartPos
1	,Y	No	UMC09,UMC11 RE-AS,TL	UMASOIL	43	2	1

- Preparation Info ---

Report As, Tl
KP 4/10/96

t#	Uptake	Uptake#2	Final	Dil.Factor

Samples Prepared.

ck #1

s	Row	Col	Sample Name	Set #	#Used	Type
1	1	1	ICV1 M64-43-01	-NA-	1	QC Standard
2	1	2	STD4 M64-43-01	-NA-	1	Standard
3	1	3	STD3 M64-43-01 5mls/10mls	-NA-	1	Standard
4	1	4	STD2 M64-43-01 1ml/10mls	-NA-	1	Standard
5	1	5	STD1 M64-45-02	-NA-	1	Standard
6	1	6	STD0	-NA-	1	Standard
7	1	7	Blank	-NA-	7	Blank
8	1	8	CCV M64-51-01	-NA-	6	QC Standard

...19 Not Used)

ck #2

s	Row	Col	Sample Name	Set #	#Used	Type
1	1	1	CRII1 M64-45-02	1	-NA-	Sample
2	1	2	ICSABI1 M64-52-05	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
0	1	10	03040-03	1	-NA-	Sample
1	1	11	03040-01 5X	1	-NA-	Sample
2	1	12	03040-01D 5X	1	-NA-	Sample
3	1	13	03040-01S 5X	1	-NA-	Sample

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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

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

Appendix II

Audit Finding Report Form

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 III

Area Names

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 IV

Finding Type

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

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 inadeq./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

CLLED

Written by: Harry Romberg
Harry Romberg, Quality Assurance Officer

Date: 6-17-96

Approved by: Karen J Kotz
Karen Kotz, Laboratory Director

Date: 6/20/96

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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

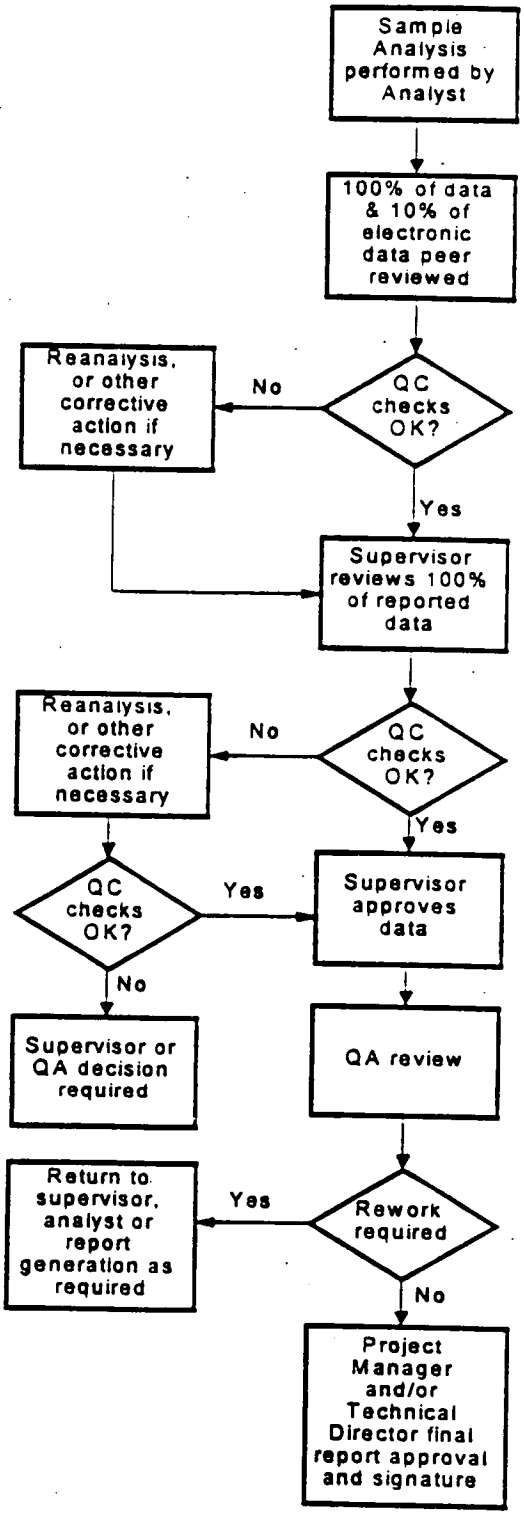
Laucks SOPs

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)

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LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1019

Title: **Controlling, Maintaining, and Monitoring Laboratory Logbooks**

Revision history:

Number	Date
0	4/11/96

Written by: Harry Romberg Date: 4-11-96
Harry Romberg, QA Officer

Approved by: Karen J Kotz Date: 4/11/96
Karen Kotz, Laboratory Director

UNCONTROLLED

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APPENDIX I 8

EXAMPLE QA LOGBOOK LABEL 8

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.

1.2 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 shall be controlled to ensure that correct documents are being used.

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.

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
- Maintain master copies of notebook pages (in instances where pre-printed pages with a specific format are used.
- 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.
- Request blank notebooks sufficiently in advance of need to allow adequate time for reproduction and binding
- 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.

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.

4.1.2 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).

4.1.3 Bound notebooks shall conform to the following:

- Binding will be of a type that will make the removal and reinsertion of pages readily noticeable
- 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 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.

4.1.4 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.

4.1.5 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 Monitoring of Laboratory Logbooks

4.4.1 The QA Officer will verify during periodic audit and surveillance activities that notebooks are properly completed and maintained.

Appendix I

Example QA Logbook Label

Laucks
Testing Laboratories, Inc.

QA Book No.:

Issued To:

Used For:

Dept. Book No.:

Start Date:

End Date:

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-2001

Title: **Waste Segregation and Disposal**

Revision history:

Number	Date
1	04/10/91
2	09/11/91
3	04/26/94
4	04/09/97

Written by: Harry Romberg — Date: 4-9-97
Harry Romberg, Quality Assurance Officer

Reviewed by: Kathy Kreps — Date: 4-9-97
Kathy Kreps, Senior Project Manager

Approved by: Karen J. Kotz — Date: 4/9/97
Karen Kotz, Laboratory Director

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1. Introduction and Scope

1.1 Method Description

1.1.1 The purpose of this SOP is to describe the laboratory waste disposal scheme currently in place at Laucks. The primary waste streams described include solvents, PCB oil wastes, COD 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 collection or 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 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.

4.1.2 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. 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.

4.1.3 In addition, corrosive and flammable waste collection drums must have a sticker which indicates their corrosive or flammable nature (see Appendix B).

4.1.4 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.

4.1.4.1 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.

4.1.5 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 5 days so QA should actually be contacted 85 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.

4.1.6 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 in the inorganics or organics instrument preparation areas but these **must** be transported to the primary drums in Extractions within 90 -days of their initial collection. After that time, when new materials are collected, the Hazardous Waste sticker should be replaced with a new sticker with a new initial collection date.

4.2.3 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.

4.2.4 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.

4.2.5 When 3 or more full 55 gallon drums of this waste have been accumulated or 85 days have passed since the beginning of collection of the oldest accumulated drum, QA must be contacted to arrange for transport and disposal.

4.2.6 At the time of this writing, Laucks uses Rollins Chempak 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 as previously noted.

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 85 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 Van Waters and Rogers (VWR) as the facility of choice for handling this waste stream. VWR handles the waste for Laidlaw Environmental. 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.

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 Van Waters and Rogers (VWR) as the facility of choice for handling this waste stream. VWR handles the waste for Laidlaw Environmental. 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 QA must be notified 85 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.

4.5.3 The only stickers these drums must have is the Hazardous Waste sticker with the date accumulation was started clearly marked.

4.5.4 At the time of this writing, Laucks uses Rollins Chempak 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.

4.6 PCB Oil Waste Disposal

4.6.1 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 contain PCBs.

4.6.2 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.

4.6.3 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 and must also send a certificate of disposal after disposal has occurred.

4.6.4 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. For this reason, it also does not fall within the state requirement for disposal within 90 days. Eastern Electric is responsible for filing appropriate reports.

5. Reports

5.1 Disposal Paperwork

5.1.1 All of our current vendors 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.

5.1.1.2 In addition, Rollins Chempak provides a copy of the manifest that must be sent by the laboratory to the California EPA, since that's where their accumulation facility is located. This applies only to waste being handled by Rollins.

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

HAZARDOUS
WASTE

STATE AND FEDERAL LAW PROHIBITS IMPROPER DISPOSAL

IF FOUND, CONTACT THE NEAREST POLICE OR PUBLIC SAFETY AUTHORITY AND THE WASHINGTON STATE DEPARTMENT OF ECOLOGY, OR THE U.S. ENVIRONMENTAL PROTECTION AGENCY.

ACCUMULATION START DATE _____ E.P.A. WASTE NO. _____

D.O.T. PROPER SHIPPING NAME _____

AND _____

U.N. OR N.A. NO. _____

GENERATOR NAME _____

ADDRESS _____

CITY _____ STATE _____

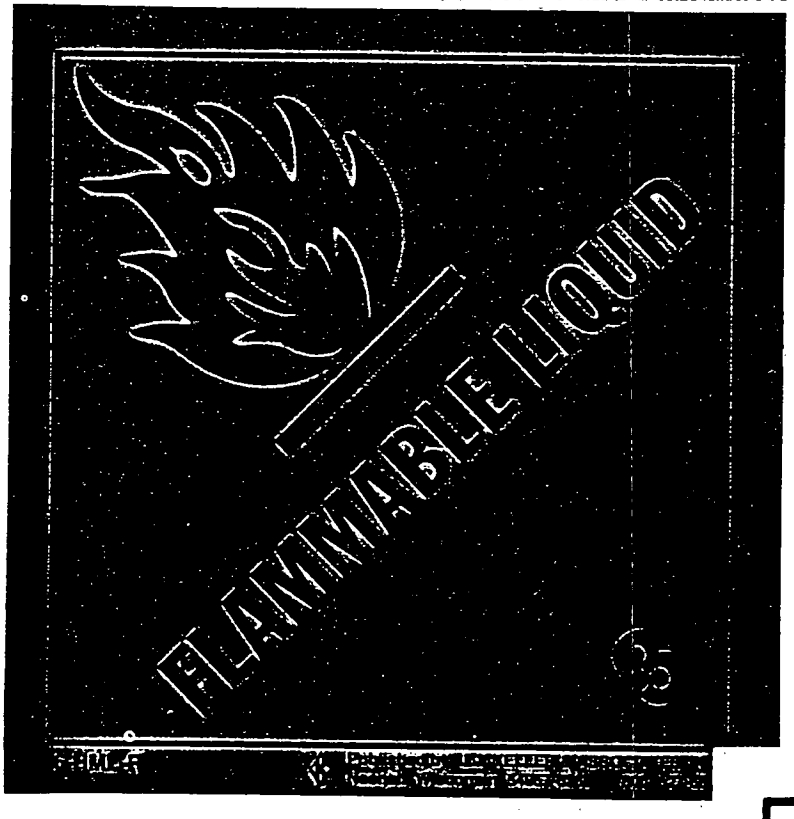
E.P.A. I.D. NO. _____ MANIFEST DOCUMENT NO. _____

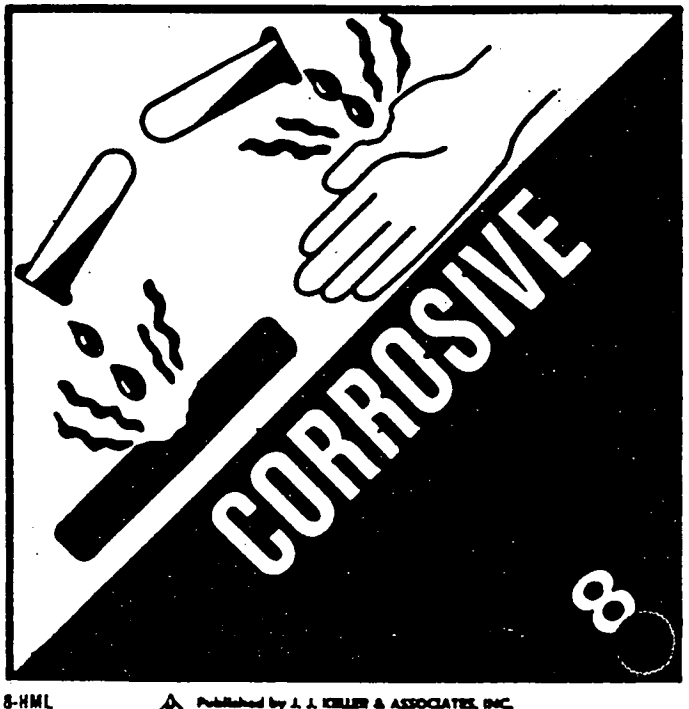
HAZARDOUS WASTE
HANDLE WITH CARE

PINACLE SYSTEMS CORP. • (206) 556-8367

Appendix II

Corrosive and Flammable Stickers





8-HML

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LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-4002

Title: **Electronic Sample Entry and Log-In**

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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

UNCONTROLLED

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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
9401497
 KEYS    ST   ORDER # 94-01-496                          RECEIVED 01/12/94   SAMPS
9401485 WR                                               DATE DUE 01/26/94     2
9401486 TR    CLIENT 1-CH-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 0
9401490 TR                          #REP/INV  3   3
9401491 WR                                               QUOTED $
9401492 WR   COMPANY C.H. Niece & Co.                    SAMPLE $ UNKNOWN    MS   4
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          ORDER# 94-04-191/WR CON MARKB                DEPTS/TESTS
9404176-12A      CLIENT PETER PAN    CAT                                      HIST1
9404176-13A     WRITTEN 04/05/94      BY HOLLYW                               HIST2
9404176-13B
9404176-13C        DASH # 02 A CATEGORY
9404176-13D    SAMPLE ID Mahi Mahi Lot #X3159 (#2)
9404176-14A    COLLECTED 04/05/94          QA/QC   MAT F
9404176-15A       STORED Analyst/Freezer  RECVD 04/05/94 SC    2
9404176-15B                                DUE 04/15/94
9404176-15C    JOB HIST2 FL   QC?         QUOTE    75.00
9404176-15D   NAME Histamine              SDG#          StID#
9404176-16A   TESTS:DEP/CAT/NAME/D/R
9404176-17A      HISTAM
9404176-17B
9404176-17C
9404176-17D
9404176-18A  -SAMPLE AND WORK DESCRIPTION-
9404191-01A   This job code is for any additional samples
9404191-02A
                                                                  CL NL SL J1
F5=Result,Price  F6=Lookup,NextJob,StateID   F10=More   Shft-Fn=Fn Help
```

FRAC Screen

4.2.5. 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 SPVMITO                             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

```
COM
  KEYS
BOG05G
BOG05I
BOG06G
BOG06I
BOG07G
BOG07I
BRPTSS
CANONS
CB2SFU
CB3-3U
CBSFXS
COM10E
COM10G
COM10I
COM10P
COM10S
COM10U
COM11E
COM11G
```

SDG Database

SDG Group : COM10 Date Due: 03/13/93 Created: 02/19/93
Fraction : E SPUMITO Updated: 02/19/93
Project : COM (EDB) Client:
SAS Number: Case Number: Max. Samps:

Work Ord	Samp Num	UTSR	Date Collected
9302598	17	02/16/93	02/13/93
9302598	20	02/16/93	02/13/93

5=Lookup,Copy,Print 6=Toggle F10=More Shft-Fn=Fn Help CL NU SL J1

SDG, Screen 2

```
COM
  KEYS
BOG05G
BOG05I
BOG06G
BOG06I
BOG07G
BOG07I
BRPTSS
CANONS
CB2SFU
CB3-3U
CBSFXS
COM10E
COM10G
COM10I
COM10P
COM10S
COM10U
COM11E
COM11G
```

SDG Database

SDG Group : COM10 Date Due: 03/13/93 Created: 02/19/93
Fraction : E SPUMITO Updated: 02/19/93
Project : COM (EDB) Client:
SAS Number: Case Number: Max. Samps:

Work Ord	Samp Num	EDB	Fractions
9302598	17	[illegible]	
9302598	20	[illegible]	

5=Lookup,Copy,Print 6=Toggle F10=More Shft-Fn=Fn Help CL NL SL J1

SDG, Screen 3

```
                                   SDG Database
COM                SDG Group  : COM10          Date Due: 03/13/93          Created:  02/19/93
  KEYS             Fraction   : E SPUMITO                                  Updated:  02/19/93
BOGO5G             Project    : COM CEDB1                 Client:
BOGO5I             SAS Number:                 Case Number:                Max. Samps:
BOGO6G
BOGO6I
BOGO7G
BOGO7I
BRPTSS
CANONS
CB2SFV
CB3-3V             Comments:
CBSFXS
COM10E
COM10G
COM10I
COM10P
COM10S
COM10V
COM11E
COM11G
                                                                          CL NO 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.

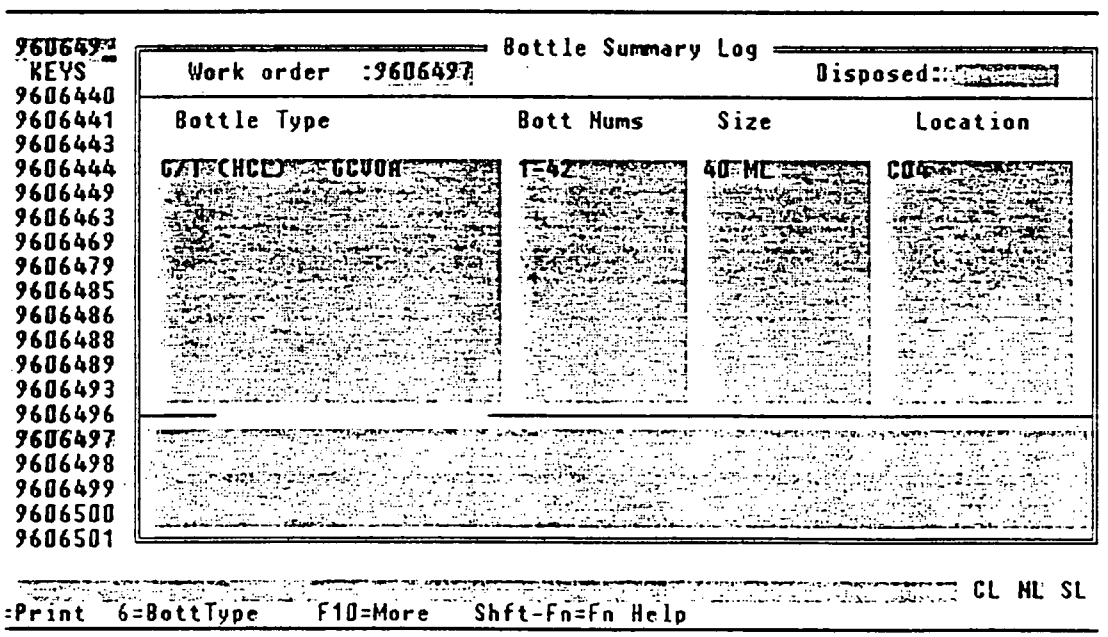
4.4.4. Storage locations

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\Hoily\docs\fax\ncmemo 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

W[illegible] 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 += 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

Project: _____ LTL Number: _____

Number of Containers (*optional*): _____

Storage Unit: _____ SDG Number (*optional*): _____

Sample Numbers	Matrix (*optional*)	Location (*shelf*)	Logged Out Date	Logged Out Time	By	Logged In Date	Logged In Time	By	Action

Samples Disposed of by _____ on _____

Laucks Testing Laboratories, Inc.

Organic Extractions Custody Log

Samples Entered By	Time	Date	JOB #	
			Client	
Samples Moved From Bldg. 940 to 921 by:			*Matrix*	*Sample #*
			SOIL	
			WATER	
			SLUDGE	
			MISC.	

Comments:

APPENDIX 5

Laucks Testing Lab pH Log Form

Laucks Testing Lab pH Log Form

Date _____

Analyst	WO Number	Fractions	pH before	Volume Acid Added/Type	Resulting pH

APPENDIX 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	Glass, Teflon-lined Septum, 40 ml capacity	Cool, 4° C, no headspace	14 days	Method 8010 GC/Hall - Direct Injection or Headspace, Method 5020 Purge-and-Trap, Method 5030
	20 grams for solids	Above or Glass, 2-4 oz. capacity	Cool, 4° C, packed to avoid headspace	14 days	or Method 8260/Method 8240, GC/MS Purge-and-Trap Method 5030
Nonhalogenated Volatile Organics	2 - 40 ml containers for liquids	Glass, Teflon-lined Septum, 40 ml capacity	Cool, 4° C, adjust pH ≤ 2 with HCl, no headspace	14 days, with preservation 7 days, if not preserved	Method 8015 GC/FID Direct Injection or Headspace, Method 5020 Purge-and-Trap, Method 5030
	20 grams for solids	Above or Glass, 2-4 oz. capacity	Cool, 4° C, packed to avoid headspace	14 days	or Method 8260/Method 8240, GC/MS Purge-and-Trap Method 5030

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

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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

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	-----

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.**

D. METALS ANALYSIS (EPA Methods for Chemical Analysis of Water and Wastes, March 1983, or APHA Standard Methods, 15 Edition. and EPA SW-846, 3rd Edition) (continued)

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Cadmium (Cd)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 213.1 or EPA 7130, Flame AA EPA 213.2 for EPA 7131, Furnace AA
Calcium (Ca)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 215.1 or EPA 7140, Flame AA
Chromium (Cr)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 218.1 or EPA 7190, Flame AA EPA 218.2 or EPA 7191, Furnace AA EPA 218.3 or EPA 7198, Chelation Extraction
Cobalt (Co)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 219.1 or EPA 7200, Flame AA EPA 219.2 or EPA 7201, Furnace AA
Copper (Cu)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 220.1 of EPA 7210, Flame AA EPA 220.2, Furnace AA
Gold (Au)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 231.1, Flame AA EPA 231.2, Furnace AA
Iron (Fe)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 236.1 or EPA 7380, Flame AA EPA 236.2, Furnace AA

** Each metal can also be analyzed by EPA 200.7, EPA 6010A, or EPA 6020.

D. METALS ANALYSIS (EPA Methods for Chemical Analysis of Water and Wastes, March 1983, or APHA Standard Methods, 15 Edition. and EPA SW-846, 3rd Edition) (continued)

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Lead (Pb)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 239.1 or EPA 7420, Flame AA EPA 239.2 or EPA 7421, Furnace AA
Magnesium (Mg)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 242.1 or EPA 7460, Flame AA
Manganese (Mn)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 243.1 or EPA 7480, Flame AA EPA 243.2 or EPA 7481, Furnace AA
Molybdenum (Mo)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 246.1 or EPA 7520, Flame AA EPA 246.2, Furnace AA
Nickel (Ni)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 249.1 or EPA 7610, Flame AA EPA 249.2, Furnace AA
Potassium (K)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 258.1, Flame AA
Selenium (Se)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 270.2 or EPA 7740, Furnace AA EPA 270.3 or EPA 7741, Hydride AA

** Each metal can also be analyzed by EPA 200.7, EPA 6010A, or EPA 6020.

D. METALS ANALYSIS (EPA Methods for Chemical Analysis of Water and Wastes, March 1983, or APHA Standard Methods, 15 Edition. and EPA SW-846, 3rd Edition) (continued)

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Silver (Ag)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 272.1 or EPA 7760, Flame AA EPA 272.2, Furnace AA
Sodium (Na)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 273.1 or EPA 7770, Flame AA
Thallium (Tl)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 279.1 or EPA 7840, Flame AA EPA 279.2 of EPA 7841, Furnace AA
Tin (Sn)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 282.1 or EPA 7870, Flame AA EPA 282.2, Furnace AA
Titanium (Ti)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 283.1, Flame AA EPA 283.2, Furnace AA
Vanadium (V)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 286.1 or EPA 7910, Flame AA EPA 286.2 or EPA 7911, Furnace AA
Zinc (Zn)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 289.1 or EPA 7950, Flame AA EPA 289.2, Furnace AA

** Each metal can also be analyzed by EPA 200.7, EPA 6010A, or EPA 6020.

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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.**

F. INORGRANIC ANALYSIS: DRINKING WATER or TITLE 22 - CAL/DOHS

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Arsenic**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	EPA 206.3, Furnace AA
Barium**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	EPA 208.1, Flame AA
Cadmium**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	EPA 213.1 , Flame AA or EPA 218.2, Furnace AA
Chromium**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	EPA 218.1, Flame AA EPA 218.2, Furnace AA
Lead**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	EPA 239.2, Furnace AA
Mercury	100 ml	Plastic or Glass	HNO_3 to pH <2	28 days	EPA 245.1 ,Cold Vapor
Selenium**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	EPA 270.2 ,Furnace AA
Silver**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	EPA 272.1 ,Flame AA
Nitrate-Nitrogen	50 ml	Plastic or Glass	Cool, 4° C add H_2SO_4 to pH <2	14 days	EPA 352.1, Brucine Sulfate
			Cool, 4° C	48 hours	EPA 353.3, Cadmium Reduction
Fluoride	300 ml	Plastic or Glass	None	28 days	EPA 340.2, Ion Selective Electrode

** Each metal can also be analyzed by EPA 200.7 or EPA 200.8.

G. GENERAL PHYSICAL ANALYSIS: DRINKING WATER or TITLE 22 - CAL/DOHS

PARAMETER	VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Color	50 ml	Plastic or Glass	Cool, 4° C	48 hours	EPA Method 110.2
Odor	200 ml	Plastic or Glass	Cool, 4° C	48 hours	EPA Method 180.1, Threshold Odor
Turbidity	100 ml	Plastic or Glass	Cool, 4° C	48 hours	EPA Method 180.1, Nephelometric

H. GENERAL WET CHEMISTRY AND MISCELLANEOUS ANALYSIS

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Acidity	100 ml	Plastic or Glass	Cool, 4° C	14 days	EPA 305.1, Titrimetric
Alkalinity	50-100 ml	Plastic or Glass	Cool, 4° C	14 days	EPA 310.1 ,Titrimetric
Ammonia Nitrogen	100 ml	Plastic or Glass	Cool, 4° C H_2SO_4 to pH <2	28 days	EPA 350.1, Colorimetric
BOD	1 liter	Plastic or Glass	Cool, 4° C	48 hours	EPA 405.1
Boron	100 ml	Plastic	None	28 days	EPA 212.3 ,Curcumin
Chloride	100 ml	Plastic or Glass	None	28 days	EPA Method 325.3, Mercuric Nitrate or EPA 300.0, Ion Chromatography
COD	20 ml	Plastic or Glass	Cool, 4° C H_2SO_4 to pH <2	28 days	EPA Methods 410.4, Colorimetric
Coliform Fecal Coliform	100 ml	Sterilized Plastic Bottles	Cool, 4° C $Na_2S_2O_3$ preserved for presence of free chlorine	6 hours or 30 hours depending on the test requested	Standard Method 909A or 909C
Color	50 ml	Plastic or Glass	Cool, 4° C	48 hours	EPA 110.2 or 110.3
Cyanide	500 ml	Plastic or Glass	Cool, 4° C NaOH to pH >12	14 days	EPA 335.3, Colorimetric
Electrical Conductivity	50 - 100 ml	Plastic or Glass	Cool, 4° C	28 days	EPA 120.1, EC Meter
Fluorides	300 ml	Plastic or Glass	None	28 days	EPA 340.2, Ion Specific Electrode or EPA 300.0, Ion Chromatography
MBAS	500 ml	Plastic or Glass	Cool, 4° C	48 hours	EPA 425.1, Colorimetric

H. GENERAL WET CHEMISTRY AND MISCELLANEOUS ANALYSIS (continued)

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Nitrate Nitrogen	100 ml	Plastic or Glass	Cool, 4° C	14 days	EPA 300.0, Ion Chromatography
Nitrate Nitrite Nitrogen	100 ml	Plastic or Glass	Cool, 4° C Add H_2SO_4 to pH <2	14 days	EPA 353.3, Cadmium Reduction
Nitrite Nitrogen	50 ml	Plastic or Glass	Cool, 4° C	48 hours	EPA 354.1, SPectrophotometric
Odor	200 ml	Plastic or Glass	Cool, 4° C	24 hours	EPA 140.1
Oil and Grease	1 liter	Glass	Cool, 4° C Add H_2SO_4 to pH <2	28 days	EPA 413.1, Gravimetric
Orthophosphate	50 ml	Plastic or Glass	Cool, 4° C	48 hours	EPA 365.2, Ascorbic Acid
pH	50 ml	Plastic or Glass	None	Immediate	EPA Method 150.1, pH Meter
Phenolics	500 ml	Glass	Cool, 4° C Add H_2SO_4 to pH <2	28 days	EPA 420.1, 4-AAP
Radioactivity	1-18 liters	Plastic or Glass Glass only for Tritium	HCl or HNO_3 * to pH <2 *Some exceptions	---	Standard Method 701
Silica	50 ml	Plastic	Cool, 4° C	28 days	EPA 370.1, Colorimetric Flame AA Method
Sulfates	50 ml - 100 ml	Plastic or Glass	Cool, 4° C	28 days	EPA 375.4, Turbidimetric or EPA 300.0, Ion Chromatography
Sulfides	500 ml	Plastic or Glass	NaOH to pH >9 2 ml Zinc Acetate Cool, 4° C	7 days	EPA 376.1 ,Titrimetric
Sulfites	50 ml	Plastic or Glass	None	Immediate	EPA 377.1 ,Titrimetric
TOC	25 ml	Plastic or Glass	Cool, 4° C H_2SO_4 to pH <2	28 days	EPA 415.2, TOC Analyzer
Total Dissolved Solids	100 ml	Plastic or Glass	Cool, 4° C	7 days	EPA 160.1, Gravimetric

H. GENERAL WET CHEMISTRY AND MISCELLANEOUS ANALYSIS (continued)

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Total Hardness	50-100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	EPA 130.2, Titrimetric Standard Method 314-A Calculation
Total Kjeldahl Nitrogen	1 liter	Plastic or Glass	Cool, 4° C Add H_2SO_4 to pH <2	28 days	EPA 351.4, Ion Specific Electrode
Total Organic Nitrogen TON-TKN-NH_3-N	1 liter	Plastic or Glass	Cool, 4° C Add H_2SO_4 to pH <2	28 days	EPA 351.4 and EPA 350.3
Total Petroleum Hydrocarbons Scan, by GC	400 ml to 1 liter Approximately 50 grams for a solid sample	Glass	Cool, 4° 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

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APPENDIX III **11**

EXAMPLE OF SDG PAPERWORK 11

1. Introduction and Scope

1.1. Method Description

1.1.1 The project manager (PM) or their designee reviews the LIMS computer entry versus the chain-of-custody (COC) for accuracy. This should be done as soon as practical and within twenty-four hours (24) of sample receipt.

1.1.2 This method is restricted to use by the person (usually a project manager) who performed the work order transmittal.

1.2. Definition of Terms

1.2.1. SDG - Sample Delivery Group

Projqc - Project QC section of the LIMS. This is where all of the work orders for a specific project are listed with additional information about the QC requirements, TATs and deliverables.

2. Equipment List

2.1. Equipment

2.1.1. The person performing this procedure must have access to a computer which is connected to the LIMS.

3. Operation procedures

3.1. Reviewing the SDG Entry in LIMS

3.1.1 Once a work order has been transmitted, go to the SDG section of the LIMS. This is done by entering [SDG, space, SDG name] at the J(1): prompt. The SDG name is found in the fractions sections of the work order in the SDG field labeled 'SDG#'. **Example** - J(1): SDG HCST4. See Appendix I for an example.

3.1.2 In the SDG verify that all the samples have been entered and that the correct 'fraction' of the SDG (I,G, V, P, etc.). The letters refer to the type of analysis, i.e., Inorganics, GC, Volatiles, and PAHs. These are a few of the types of

fractions which might be contained in a SDG. It is not important to know the abbreviations. However, it should be verified that all analyses for each sample can be found in the SDG by looking at the various fractions for that SDG. A fraction may contain more than one analysis. For instance an 'I' fraction (inorganics) might have TOC, Metals and TSS.

3.1.3 When the SDG name is entered at the J(1): prompt, the 'first' page of the first fraction of a SDG will be displayed. The first fraction is determined alphabetically based upon the letter associated with an analysis. For instance, if a work order has analyses for GC analysis (G), metals (I) and PAHs (P), the fractions of a SDG would be listed in LIMS with GC analyses first, metals second, and PAHs last. The information contained on the first page is:

- Laucks sample ID
- QC Designations
- Client ID
- Matrix

3.1.4. Verify that all the samples for that fraction (analysis) have been entered. this is facilitated by entering [F6] which displays the second , third and fourth pages of a fraction. [F6] is a toggle key which pages through the four screens of each SDG entry. The far left hand column of the computer screen will always display the lists of the SDG names with the fractions appendix letter. **Example** - GSI01G (GC fraction), GSI01I (inorganics fractions). See pages 1,2 and 3 of Appendix 1 for examples of each page of a SDG.

3.1.5 The second page of a SDG contains the Laucks samples ID, the VTSR (Verified Time of Sample Receipt) and date collected information.

3.1.6 The third page of a SDG contains the Laucks sample ID and the specific analysis requested. It is important to review this page carefully. If there are many samples in that fraction, they may not all be visible at once. Move down the screen to view all samples by repeatedly pushing the down arrow key, [↓].

3.1.7 Once this fraction has been checked, the other fractions can be checked by using the [F3] (moves the cursor up) and [F4] (moves the cursor down) function keys to place the cursor on the next fraction of the SDG. It is displayed in the column on the left of the screen.. It is possible to view the same page of different fractions by just moving the cursor to the next fraction. For example, if you are in page three of a fraction, when you move to the next fraction, page three of this fraction will

be displayed. **[F6] controls the page selection of the SDG, [F3] and [F4] respectively in the far left column.**

3.1.8 If corrections are necessary, return the COC and log-in documents to the Sample Control department. If the person performing the review makes the corrections, the changes must be saved by entering [F8].

3.1.9 To exit the SDG, enter [F1] until the J(1): prompt is displayed.

3.2 Entering a Work Order into 'Projqc' of LIMS

3.2.1 At the J(1): prompt, enter [projqc, space, projqc name]. Example - J(1): projqc OHM_Hawaii. If you are not sure of the projqc name, a name may be entered which is similar. This will bring up the projqc in the vicinity of the name which is desired. The cursor is moved up, [F3], or down, [F4}, to the desired name. The projqc names will be displayed in the far left column of the screen. See page 1 of Appendix II.

3.2.2 Once the correct project name is displayed, move the cursor down to the next empty line in the work order column. enter the work order number. The SDG will appear in the SDG list column. Move the cursor to the matrix columns (W = Water, S = Soil, O = Oil). Under the appropriate column, enter the number of samples for each matrix. If the SDG is to remain open, no other information should be added. Enter [F8] to save the updated SDG.

3.2.3 It the SDG is to be closed, the due date for the data to be submitted to the reporting department and the due date for the hardcopy report to the client must be entered into the appropriate columns. These dates are displayed in Projqc as 'Office' and 'Client' respectively. See page 1 of Appendix II. Enter [F8] to save these changes.

3.2.4 SDG closure is determined by several factors:

- The number of samples in the SDG, per the EPA definition, should not contain more than twenty samples of the same matrix received over a period of not more than 14 calendar days.

- If it is known that more samples will be arriving for the same project in less than fourteen days, and the SDG is not full, it may be desirable to maintain the SDG in 'open' status until arrival of the next samples.

- If the client desires a fast TAT for the hardcopy package, the SDG should be closed regardless of the number of samples in it.

These are judgment decisions with the exception of the EPA definition of an SDG), which must be made by the project manager.

3.2.5 Once the SDG has been closed, send e-mail notification of the closure to all departments affected. This would be any department involved with the analyses for the work order, Sample Control and the reporting department.

3.2.6 Finally, the paperwork is submitted to the reporting department detailing the nature and status (open or closed) of an SDG. The forms submitted would be the following, arranged in the order listed below:

- Pre-package checklist
- Chain-of-Custody forms (original or top, white copy)
- * Sample Receipt Log (1) CLP (original)
- * Supplemental Sample Receipt Log (original)

* A copy of each of these forms must be made and attached to the copy of the COC for the transmittal.

See Appendix III for examples of these forms.

3.3 Creating a New Projqc

3.3.1 When a new project commences, it may be necessary to create a new entry in Projqc. The factors which would determine if this is necessary may include the following:

- The complexity of the project
- The duration of the project
- How many laboratory departments are affected by the project

Projqc entries are applicable to both CLP and non-CLP project. The information contained in this section of the LIMS is accessible by all laboratory staff who have rights to the LIMS and is a valuable form of communication for project requirements. However, it does not take the place of kick-off meetings for projects or e-mail regarding specific samples problems.

3.3.2 To create a new projqc entry, enter [projqc] at the J(1): prompt. This will display the first projqc entry in the LIMS. The cursor will be in the field called 'Proj Name:'. Enter the name of the new projqc entry. this name may consist of up to ten characters, alpha and numeric, and one underscore in the place of a hyphen, if desired. Once entered, push [Enter]. The cursor will move to the next field, 'Client Code'. See page 1 of Appendix II.

3.3.3 In the 'Client Code' field, enter the correct client code for this work order. This can be found in the work order. Push [Enter]. The cursor will next appear in the 'Desc:' field.

3.3.4 In this field enter a brief description (one cryptic sentence) of the project. Push [Enter]. the cursor will next appear in the bottom line of the projqc header. Enter the correct information for these questions (QAPP?, pH/Temp?). This would usually be 'yes' if the project is CLP or USACE (US Army Corps of Engineers).

3.3.5 Due to software complications, in the field 'Sort by (W/S):' it is best to enter [N].

3.3.6 Enter [F8] to save this new projqc. Item 3.3.1 through 3.3.5 would be considered the header information for a projqc. This is page 1 of the projqc. It is here that the work orders for that project will be entered when the samples arrive at the laboratory.

3.3.7 Next go to page 2 of the projqc. This is accomplished by pushing [F6]. The header information will already be there. The information which must be entered here is:

- Results TAT (Fax)
- Report TAT (hardcopy)
- Deliverables - CLP, LTL (Laucks Testing Laboratory or non-CLP)
- MPR - Monthly Progress Report (required for HAZWRAP projects)
- EDD deliverables - CLP or LTL (any non-CLP, custom EDD)
- Deliverables Comments - Any special project comments
- Penalties - enter any applicable penalties here

Enter [F8] to save this information.

3.3.8 Next go to page three of the projqc. This is accomplished by entering [Ctrl,F6]. of a project can be written as an overview. See page three of Appendix II. The information contained here is:

- Client name
- Project Name
- Overview
- Schedule
- Analytes
- Protocol
- QC
- Turnaround
- Penalties
- CRDLs
- Holding Times
- Deliverables
- Additional Comments

After entering this information, enter [F8] to save it. It may be advantageous to enter [F8] periodically as the information is entered to prevent loss in the event of a power surge.

Appendix I

Example of SDG Entry in LIMS

GSI01
KEYS
GLS08S
GLS08T
GLS08V
GLS09E
GLS09I
GLS09O
GLS09P
GLS09S
GLS09T
GLS09V
GSI01G
GSI01I
GSI01P
GSI01V
GSI02G
GSI02I
GSI02P
GSI02V
GSI03G

SDG Database

SDG Group : GSI01 Date Due: 11/10/95 Created: 10/12/95
Fraction : G SPVMITO Updated: 10/23/95
Project : AFCEE/Chanute 952 Client: GSI (TPH 8015) JP4
PROJQC ID : GS_CHANUTE Max. Samps: 21

Work Ord	Samp Num	QC	Client ID	Mat	TS	pH	Associated Blank
9510423	01		B952/SB-952-1/SS4,6-8/LK	S	88		B1018GSVSLW
9510423	02		B952/SB-952-1/SS9,16-18/LK	S	89		B1018GSVSLW
9510423	03		B952/SB-952-2/SS4,6-8/LK	S	83		B1018GSVSLW
9510423	04		B952/SB-952-2/SS5,8-10/LK	S	84		B1018GSVSLW
9510434	01		B952/SB-952-10/SS8,14-16LK	S	84		B1018GSVSLW
9510434	02		B952/SB-952-3/SS3,4-6/LK	S	84		B1018GSVSLW
9510434	03	MS	B 52/SB-952-3/SS6,10-12/LK	S	89		B1018GSVSLW
9510434	05	BMS	CH-5092500-S-SPOON-ERB-1	W			B1017GSVWLQ2
9510434	06		CH-POT-FB-1	W			B1017GSVWLQ2
9510434	07		CH-ASTM-5092500-FB-1	W			B1017GSVWLQ2
9510459	01		B952/SB-952-4/SS4,6-8/LK	S	84		B1018GSVSLW
9510459	02		B952/SB-952-4/SS7,12-14/LK	S	89		B1018GSVSLW
9510459	03		B952/SB-952-5/SS4,6-8/LK	S	87		B1018GSVSLW
9510459	04		B952/SB-952-5/SS6,10-12/LK	S	87		B1018GSVSLW
9510459	08		B952/SB-952-6/SS4,6-8/LK	S	84		B1018GSVSLW
9510459	09		B952/SB-952-6/SS6,10-12/LK	S	90		B1018GSVSLW
9510497	01		B952/SB-952-7/SS3,4-6/LK	S	89		B1018GSVSLW
9510497	02		B952/SB-952-7/SS8/4-16/LK	S	91		B1018GSVSLW
9510540	01		B952/SB-952-8/SS3,4.0-6.0	S	90		B1018GSVSLW
9510540	02		B952/SB-952-8/SS6,10.0-12	S	90		B1018GSVSLW

SDG Database Report
Page 2

Laucks Testing Labs Report Date: 01/25/96

SDG Group : GSI01 Date Due: 11/10/95
Fraction : G
Project : AFCEE/Chanute 952 Client: GSI (TPH 8015) JP4
SAS Number: _____ Case Number: _____

Work Ord	Samp Num	VTSR	Date Collected
9510423-01		10/11/95	10/10/95
9510423-02		10/11/95	10/10/95
9510423-03		10/11/95	10/10/95
9510423-04		10/11/95	10/10/95
9510434-01		10/12/95	10/11/95
9510434-02		10/12/95	10/11/95
9510434-03		10/12/95	10/11/95
9510434-05		10/12/95	10/10/95
9510434-06		10/12/95	10/10/95
9510434-07		10/12/95	10/10/95
9510459-01		10/13/95	10/11/95
9510459-02		10/13/95	10/11/95
9510459-03		10/13/95	10/11/95
9510459-04		10/13/95	10/11/95
9510459-07		10/13/95	10/12/95
9510459-08		10/13/95	10/12/95
9510459-09		10/13/95	10/12/95
9510497-01		10/14/95	10/12/95
9510497-02		10/14/95	10/12/95
9510540-01		10/17/95	10/15/95
9510540-02		10/17/95	10/15/95

SDG Database Report
Page 3

Laucks Testing Labs

Report Date: 01/25/96

SDG Group : GSI01 Date Due: 11/10/95
Fraction : G
Project : AFCEE/Chanute 952 Client: GSI (TPH 8015) JP4
SAS Number: _____ Case Number: _____

Work Ord	Samp Num	Fractions									
		JP4									
9510423-01		X									
9510423-02		X									
9510423-03		X									
9510423-04		X									
9510434-01		X									
9510434-02		X									
9510434-03		X									
9510434-05		X									
9510434-06		X									
9510434-07		X									
9510459-01		X									
9510459-02		X									
9510459-03		X									
9510459-04		X									
9510459-07		X									
9510459-08		X									
9510459-09		X									
9510497-01		X									
9510497-02		X									
9510540-01		X									
9510540-02		X									

Comments:

SDG Database

G
KEYS
GLS08O
GLS08P
GLS08S
GLS08T
GLS08V
GLS09E
GLS09I
GLS09O
GLS09P
GLS09S
GLS09T
GLS09V
GSI01G
GSI01I
GSI01P
GSI01V
GSI02G
GSI02I
GSI02P

SDG Group : GSI01 Date Due: 11/10/95 Created: 10/12/95
Fraction : I SPVMITO Updated: 10/17/95
Project : AFCEE/Chanute 952 Client: GSI (Inorganic)
PROJQC ID : GS_CHANUTE Max. Samps: 21

Comments:
Use Control F6 for more PROJQC details.

F7=Del,/n,Name 8=Write,/n,In_use 9=Print,Db_enter?,Keys? 10=More,Help,Key

Appendix II

Example of Projqc in LIMS

OHM
KEYS
HC_MANANN
HC_MANANNS
HC_MANANNW
KIC_ARCO
LOW_AFB_IN
LOW_AFB_OR
MCCHORDAFB
MCCHORDLTM
METRO_TBT
OHM-HAWAII
OHM-SITEA
OHM-SITEF
PASCO_AIR
PASCO_P
PASCO_S
PASCO_W
Q_CITY_F
SKAGIT
SODASPRING

Project QC Requirements Database

Proj Name: OHM-HAWAII Client Code: OHM_HAWAII Created: 12/19/95
Desc: UST sites Updated: 01/22/96
QAPP? Y pH/Temp? Y Sort by (W/S): N By : DIANA

Ord#	Last Date	SDG#	Office	Client	Matrix W	Matrix S	Matrix O
9512516	12/19/95	HI033	01/31/96	02/02/96	2	3	
9601349	01/22/96	HI034	02/20/96	02/22/96	1	7	
9601351	01/10/96	HI035			3		
9601587	01/22/96	HI034	02/20/96	02/22/96	2	6	

F7=Del,,Name 8=Write,,In_use 9=Print,Help menu,PrtSc 10=More,Help,Key

OHM
KEYS
HC_MANANN
HC_MANANNS
HC_MANANNW
KIC_ARCO
LOW_AFB_IN
LOW_AFB_OR
MCCHORDAFB
MCCHORDLTM
METRO_TBT
OHM-HAWAII
OHM-SITEA
OHM-SITEF
PASCO_AIR
PASCO_P
PASCO_S
PASCO_W
Q_CITY_F
SKAGIT
SODASPRING

Project QC Requirements Database

Proj Name: OHM-HAWAII Client Code: OHM_HAWAII Created: 12/19/95
Desc: UST sites Updated: 01/22/96
QAPP? Y pH/Temp? Y Sort by (W/S): N By : DIANA

Results turnaround 7 WORK.DAYS
Report turnaround 30 CAL.DAYS

Deliverables		Penalties
LTL report		
"CLP" report	X	
MPR required		
CLP disk		
LTL disk		
Other		
Other		

Deliverables comments

F7=Del,,Name 8=Write,,In_use 9=Print,Help menu,PrtSc 10=More,Help,Key

File: Y:OHM-HAWAII.PCC
Date: 01/24/96

Client : OHM Remediation Services, Inc.

Project Name: UST Sites in Hawaii

Overview : 90% project consists of Soil samples, 10% water/rinsate samples

Schedule : Delivery of samples over 6 months. Weekly schedule will be obtained one week in advance of sample receipts.

Analytes : Water
29- BTE samples via 8020
36- 8015 Mod (TPH gas or diesel or 418.1)
17- 6010 for Pb, Cd, Cr
30- 8310 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,SO

Turnaround : FAX 7 working days from sample receipt Hard Copy 30 calendar days from SDG closure EDD Not applicable

File: Y:OHM-HAWAII.PCC
Date: 01/24/96

Penalties : See PROJQC, page 2

CRDLs : Yes! See minimum detection limits in handout submitted to; JMB,MN,Bill,BarbM,MC,CD,MS,MIK,PJ,TM,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)
9600312	1/16/96

Due to Office: 2/7/96

Due to client: 2/9/96

E.D.D.: yes ☐ Custom or CLP *(circle one)* no ☑

If yes, can the package ship without the disk? _____

Full Deliverables? yes ☑ no ☐

If no, what forms/raw data are required? _____

No. of Copies: 1

Special package requests:

Assigned Document controller: _____

THIS INFORMATION WILL BE USED FOR REPORTING/BILLING* (SEE BELOW)

NAME: Groundwater Technology GSI
ADDRESS: 19033 W Valley Hwy D-109
Kent WA 98302
ATTENTION: Diana Spence
PROJECT NAME: Bangor Site A
PROJECT CONTACT: Stan Thompson
TELEPHONE/FAX: 360 779 9564/ 360-779-1676
JOB/P.O. NO.: 83001-2153 110
SAMPLER (SIGNATURE): (PRINTED NAME) Daryl Braun

CHAIN OF CUSTODY RECORD

Rad 1313

WORK ORDER ID# 83001-2153 PAGE 1 OF 1

DATE 1-8-96

SUBMITTED AT:

Laucks Testing Laboratories, Inc.

TESTS TO PERFORM: M8330

LAB SAMPLE	SAMPLE ID / LOCATION	DATE	TIME	NO. OF CONTAINERS	OBSERVATIONS, COMMENTS, SPECIAL INSTRUCTIONS
1)	INF-CL-209-010896	1-8-96	0900	2L	Fax Results to 360-779-16

INSTRUCTIONS
1. USE ONE LINE PER SAMPLE.
2. BE SPECIFIC IN TEST REQUESTS.
3. CHECK OFF TESTS TO BE PERFORMED FOR EACH SAMPLE.

*BILLING INFORMATION, IF DIFFERENT THAN ABOVE
NAME
ADDRESS
ATTN:
CITY, STATE, ZIP

TURNAROUND REQUEST
- [ ] 24-48 HRS (100% SUR)
- [x] 5 DAYS (50% SUR)
- [ ] STD. 10-11 DAYS
- [ ] OTHER

TOTAL NO. OF CONTAINERS 2L

CHAIN OF CUSTODY SEALS? [ ] YES [ ] NO [ ] NA

SHIPPED VIA: [ ] UPS [ ] FED-EX [ ] BUS [ ] HAND [ ] ____

TEMPERATURE ____

[ ] CLIENT [ ] REPRESENTATIVE

RELINQUISHED BY (SIGN AND PRINT)	DATE / TIME	RECEIVED BY (SIGN AND PRINT)	DATE / TIME
Daryl Braun	1-8-96 1600	Pony Express	
		PAM R. JOHNSON LAUCKS TESTING LABS	1/9/96 0726

FINAL REPORT COPY

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

P:\PM\Holly\docs\samplememo.doc

Initial once samples are checked in _____

DATE RECEIVED: 1/9/96
TIME RECEIVED: 0726
CLIENT NAME: GSI
SDG #: GTE27
COC #: N/A

SAMPLE LOG-IN DATE: 1/9/96
WORKORDER #: 9601313
CLIENT PROJECT: 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 20/23	2)7								

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

TEMPLOG.DOT : 07/1/95

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

UNCONTROLLED

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Appendix I 10

1. Introduction and Scope

1.1 This SOP descibes the contents of the different levels of reports at Laucks Testing Laboratories, Inc.

1.2 Complete CLP-type data package (level IV):

A complete data package (level IV) submitted to a client consists of a cover page, a narrative, chain-of-custody copies, an index, and a separate section for each analytical fraction containing all forms and raw data. The entire package is paginated sequentially beginning with #1.

1.3 Forms only data package (level III):

A "forms only" data package (level III) submitted to a client contains all of the above with the exception of the raw data.

1.4 Database Report (level II):

A database report (level II) contains forms generated from the database and includes many of the forms in a level III or IV package. The contents are indicated in Appendix I and are not described further.

1.5 Paper Job Report (level I):

A "paper job" report is created for special chemistry and food chemistry and usually contains a format specified by the client or results only. The contents are indicated in Appendix I and are not described further.

2. Description of Contents of Level III and IV packages

Detailed below are the elements that may be included in a level III or level IV package. Appendix I contains tables that signify whether an element is incorporated into a final report. Client specific requests may dictate that some elements may be added or deleted and these are documented during project initiation in the LIMs system.

2.1 Cover Page

The package cover page will contain the laboratory name, client name, work order numbers, SDG number and the date.

2.2 Narrative

2.2.1 Sample receipt and Identification:

This section lists all client sample names, the corresponding laboratory sample names and the analyses requested for each sample. (The analyses are generally abbreviated to three letters.)

2.2.2 Analytical request key:

This section defines the abbreviations listed in the above section.

2.2.3 Sample Identification on Forms:

This sections is used to explain any abbreviations to client sample names on any of the forms (occasionally forms software does not accommodate lengthy client sample I.D.s).

2.2.4 General remarks on organic analysis:

These are stock comments contained in the narrative template describing general analysis conditions for each of the requested organic fractions.

2.2.5 Specific remarks on organic analysis:

These are comments written for each organic analytical fraction describing any anomalies, deviations from the specified method, dillutions, holding time violations, corrective actions etc. These comments are written by the respective analysts.

2.2.6 General remarks on inorganic analysis

These are stock comments contained in the narrative template describing general analysis conditions for each of the requested inorganics fractions.

2.2.7 Specific Remarks on inorganic analysis:

These are comments written for each inorganic analytical fraction describing any anomalies,deviations from the specified method, dilution's, holding time violations,corrective actions etc. These comments are written by the respective analysts.

2.2.8 Release of data:

This page is signed by both the respective project manager and the technical director. also contained on this page is information on who to contact regarding specific questions as well as the laboratory telephone and fax numbers

2.3 Chain-of-Custody Copies:

This section contains the chains of custody received with the samples as well the laboratory receipt and temperature logs.

2.4 Index:
The index should list all data fractions and sub-fractions with the corresponding page numbers.

2.5 Organic fractions

Level IV Organic data packages are subdivided into five sections: QC Summary, Sample Data, Standards Data, Raw QC Data, and Bench Sheets.

Level III (forms only) organic data packages contain only the **forms** from these sections. No bench sheets or raw data are provided.

Only Volatiles, Semi-volatiles and Pesticides/PCBs have official CLP form numbers and protocol. Every effort is made to ensure that the same information appears on forms for all other fractions.

NOTE: the form numbers that appear below are seen only on forms for Volatiles, Semi-Volatiles and Pesticides/PCBs. Forms for all other fractions contain the same information but no actual form numbers.

2.5.1 QC Summary:

The QC Summary contains the following forms:

a.) Form II: Surrogate recovery report
b.) Form III: MS/MSD Recovery report
c.) Blank spike report
d.) Form IV: Method blank summary
e.) Form V: Tuning and Mass Calibration Standard
f.) Form VIII: Internal standards Area Summary

2.5.2 Sample Data:

Sample data contains the following forms and data

a.) Form I (analysis data sheet) including TICs
b.) Raw Data

2.5.3 Standards Data:

The standards data below are divided into two formats: Volatile/Semi-Volatile and Pesticide/PCB. Every effort is made to ensure that forms for all other fractions adhere closely to whichever of the two formats is most applicable.

2.5.3.1 Volatile/Semi-Volatile

a.) Form VI and Initial Calibration Data
b.) Form VII and Continuing Calibration Data

2.5.3.2 Pesticide/PCB

a.) Form VIII: Pesticide Analytical Sequence
b.) Form IX: Pesticide/PCB Standards Summary
c.) Form X: Pesticide/PCB Identification (positive results)
d.) Pesticide standard chromatograms and data system printouts for Evaluation of standard mix A,B, and C
e.) Pesticide standard chromatograms and data system printouts for individual standard mix A, and B
f.) Pesticide Standard Chromatograms and data system printouts for all multi-response pesticides/PCBs and quantitation standards
g.) A copy of the computer reproduction or strip chart recorder output covering the 100 fold range

2.5.4 Raw QC Data:

The Raw QC Data below are divided into three formats: Volatile, Semi-Volatile, and all other fractions.

2.5.4.1 Volatile
- a.) BFB
  - 1.) Bar graph spectrum
  - 2.) Mass listing
  - 3.) RIC: Reconstructed Total Ion Chromatogram
- b.) Blank Data
  - 1.) Form I including TICs
  - 2.) Raw data
- c.) Matrix Spike Data
  - 1.) Form I
  - 2.) Raw data
- d.) Matrix Spike Duplicate Data
  - 1.) Form I
  - 2.) Raw data

2.5.4.2 Semi-Volatile
- a.) DFTPP
  - 1.) Bar graph spectrum
  - 2.) Mass listing
  - 3.) RIC: Reconstructed Total Ion Chromatogram
- b.) Blank Data
  - 1.) Form I including TICs
  - 2.) Raw data
- c.) Matrix Spike Data
  - 1.) Form I
  - 2.) Raw data
- d.) Matrix Spike Duplicate Data
  - 1.) Form I
  - 2.) Raw data

2.5.4.3 All other fractions
- a.) Blank Data
  - 1.) Form I
  - 2.) Raw data

b.) Matrix Spike Data
  1.) Form I
  2.) Raw data
c.) Matrix Spike Duplicate Data
  1.) Form I
  2.) Raw data

2.5.5 Bench Sheets:

The bench sheets section contains any miscellaneous paper work such as log book pages, in-house tracking sheets, in-house chains of custody, extraction bench sheets etc.

2.6 Inorganics Fractions:

There are two kinds of inorganics fractions: Metals and Miscellaneous Inorganics (conventionals).

2.6.1 Metals

2.6.1.1 Cover Page:

This page lists all samples analyzed and is signed and dated by the analyst

2.6.1.2 Inorganics Analysis Data Sheet
a.) forms I

2.6.1.3 Quality Control Data
a.) Form II (part 1): Initial and Continuing Calibration Verification
b.) Form II (part 2): CRDL Standard for AA and ICP
c.) Form III: blanks
d.) Form IV: ICP Interference Check Sample
e.) Form V (part 1): Spike Sample Recovery
f.) Form V (part 2): Post Digest Spike Sample Recovery
g.) Form VI: Duplicates
h.) Form VII: Laboratory Control Sample
i.) Form VIII: Standard Addition Results
j.) Form IX: ICP Serial Dilutions
k.) Form XIII: Preparation Log
l.) Form XIV: Analysis run Log

2.6.1.4 Quarterly Verification of Instrument Parameters

a.) Form X: Instrument Detection Limits (quarterly)
b.) Form XI (part 1): ICP Inter element Correction Factors (annually)
c.) Form XI (part 2): ICP Inter element Correction Factors (annually)
d.) Form XII: ICP Linear Ranges (quarterly)

2.6.1.5 Raw Data

a.) ICP
b.) Graphite Furnace
c.) Mercury
d.) Cyanide

2.6.1.6 Digestion and Distillation Logs

a.) ICP
b.) Graphite Furnace
c.) Mercury
d.) Cyanide

2.6.2 Miscellaneous Inorganics

2.6.2.1 Cover Page

This page lists all samples analyzed and is signed and dated by the analyst.

2.6.2.2 Inorganics Analysis Data Sheet

2.6.2.3 Quality Control Data

a.) Preparation blanks database Report
b.) MS/MSD Report
c.) MS/Duplicate Database
d.) SRM Report

2.6.2.4 Raw Data

The Miscellaneous Inorganics raw data is divided into sections by analytes.

Appendix I

Laucks Testing Laboratories
Organics
Levels of Reporting

	Level I	Level II	Level III	Level IV = CLP
Narrative			Y	
Chain-of-Custody	Y	Y	Y	Y
Method Reference	Y	Y	Y	Y
Results	Y	Y	Y	Y
Surrogate Rec.		Y	Y	Y
Method Blank		Y	Y	Y
Blank Spike		Y	Y	Y
MS/MSD		Y	Y	Y
Tune			Y	Y
Initial Calib.			Y	Y
Cont. Calib.			Y	Y
IS Area				Y
Raw Data			Optional	Y
Chromatograms			Optional	Y
Bench Sheets				Y

Y indicates that this element is present in the hardcopy delivered to the client.

Laucks Testing Laboratories
Metals
Levels of Reporting

	Level I	Level II	Level III	Level IV = CLP
Narrative			Y	Y
Chain-of-Custody	Y	Y	Y	Y
Method Reference	Y	Y	Y	Y
Results	Y	Y	Y	Y
Method Blank		Y	Y	Y
SRM/LCS/BS		Y	Y	Y
MS/Dupe		Y		Y
MS/MSD			Y	Y
ICV			Y	Y
ICB			Y	Y
CCV			Y	Y
CCB			Y	Y
CRA			Y	Y
CRI			Y	Y
Post Spike			Y	Y
GFAA MSA			Y	Y
ICP Ser. Dil'n.			Y	Y
IDLs			Y	Y
Interelement Corr. Factors				Y
Linear Range				Y
Prep Log				Y
Run Log				Y
Raw Data				Y
Bench Sheets				Y

Y indicates that this element is present in the hardcopy delivered to the client.

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 Date: 4/9/97
Bill Lundberg, Inorganics Supervisor

Reviewed by: Harry Romberg Date: 4-9-97
Harry Romberg, QA Officer

Approved by: Karen J. Kotz Date: 4/9/97
Karen Kotz, Laboratory Director

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5. References 6

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.

APPENDIX B

LAUCKS TESTING LABORATORIES, INC., QUALITY ASSURANCE PLAN
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APPENDIX C

BROWN & ROOT ENVIRONMENTAL STANDARD OPERATING PROCEDURES

SOP NUMBER	Title
• CT-03	Data Validation
• CT-05	Database Records and Quality Assurance
• SF-1.6	Analysis For Volatile Compounds Using Purge-and-Trap Gas Chromatography (Field GC Analysis)

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1.0 CLP ORGANICS FOR SOLID AND AQUEOUS MATRICES

1.1 CLP Organics by GC/MS

1.1.1 Volatiles (USEPA CLP Statement of Work (SOW) 3/90)

1.1.1.1 Applicability

CLP 3/90 volatile methodology is used to determine organic compounds in most matrices including groundwater, sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments.

The CLP 3/90 volatile Target Compound List (TCL) includes the following substances:

Acetone	Chloromethane	Methylene Chloride
Benzene	Dibromochloromethane	4-Methyl-2-Pentanone
Bromodichloromethane	1,1-Dichloroethane	Styrene
Bromoform	1,2-Dichloroethane	1,1,2,2-Tetrachloroethane
Bromomethane	1,1-Dichloroethene	Tetrachloroethene
2-Butanone	1,2-Dichloroethene (total)	Toluene
Carbon Disulfide	1,2-Dichloropropane	1,1,1-Trichloroethane
Carbon Tetrachloride	cis-1,3-Dichloropropene	1,1,2-Trichloroethane
Chlorobenzene	trans-1,3-Dichloropropene	Trichloroethene
Chloroethane	Ethyl Benzene	Vinyl Chloride
Chloroform	2-Hexanone	Xylenes (total)

This method is based upon a purge-and-trap, gas chromatographic/mass spectrometric (GC/MS) procedure. Prior to analysis, samples must be prepared according to the SOW.

1.1.1.2 Interferences

Samples can be contaminated by diffusion of volatile organics (particularly chlorofluorocarbons and methylene chloride) through the sample container septum during shipment and storage. Associated field quality control blanks are analyzed in order to monitor this.

Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe or purging device is rinsed with reagent water between samples. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of reagent water to check for cross contamination.

If sample or matrix interferences are encountered, a secondary or alternate analytical column may be used to resolve the compounds of interest.

1.1.1.3 General Laboratory Practices

A method blank consisting of organic free water spiked with surrogates and internal standards should be analyzed immediately following each daily calibration, and also after the analysis of every high concentration sample.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) analyses should be conducted to determine the effects of sample matrix upon the compounds of interest.

1.1.1.4 Sample Preparation

A purge- and -trap procedure is performed to prepare and extract volatile compounds from samples and to introduce those compounds into the GC/MS.

For highly volatile matrices, direct injection preceded by dilution should be used to prevent gross contamination of the instrumentation. For pastes, dilution of the sample until it becomes free-flowing is used to ensure adequate interfacial area. The success of this method depends on the level of interferences in the sample; results may vary due to the large variability and complicated matrices of solid waste samples.

1.1.1.5 Data Overview Prior to Validation

Before commencing validation, the reviewer must preview the associated Chain-of-Custody (COC) reports to determine:

- If the appropriate number of samples are present in the data package and if each sample was correctly analyzed for the parameters and methods specified.
- The identity of all associated field quality control blanks and field duplicate pairs.

Because many samples may have required dilutions, re-extractions and/or re-analyses, the validator should preview the data package contents to determine which analyses represent the better quality data.

Unless specifically directed by client protocol, never annotate the laboratory data package. Before beginning evaluation, prepare working copies (i.e., photocopies) of all Form I reports (including those for samples, laboratory method blanks and MS/MSD analyses), and all laboratory quality control summary forms (including all initial and continuing calibration summary statistics).

1.1.1.6 Technical Evaluation Summary

All data evaluations must be conducted in accordance with applicable USEPA Regional protocols and/or specific client contract requirements. The applicable documents must be referenced during the data evaluation process as this S.O.P. is only intended as a general procedure for the data validation tasks.

General parameters such as Data Completeness, Overall System Performance, Chromatographic Quality, Detection Limits and Compound Identification are evaluated concurrently with the parameters discussed in the following subsections.

1.1.1.6.1 Holding Times

Holding times are evaluated by reviewing the COC reports, the individual sample Form I reports, and the associated laboratory raw data. Holding times are calculated from date of collection to date of analysis.

For unpreserved aqueous samples, generally a 7-day maximum holding time allowance for aromatic compounds, along with a 14-day maximum holding time allowance for chlorinated hydrocarbons is used.

The technical maximum holding time allowance for aqueous samples preserved with hydrochloric acid (HCl) is 14 days.

No technical holding times have been for solid matrices promulgated; a 14-day maximum holding time allowance is currently being used.

Positive results in affected samples are generally qualified as estimated (J); nondetects (UJ). These results are biased low. Some USEPA Regions apply the bias qualifiers, L and UL, instead. If the holding times are exceeded by a factor of 2 or more, the holding time exceedance is considered to be gross, and positive results are generally qualified as estimated (J); nondetects are generally considered to be unreliable and are qualified (R). Results for which the holding time was grossly exceeded are biased very low.

1.1.1.6.2 Calibration

Check that an initial calibration was performed for each instrument used for analysis and that all calibrations were performed at all appropriate concentration levels within 12 hours of the associated instrument tuning.

Review the data package Form Vs (tuning) using the applicable USEPA Regional Functional Guidelines, and qualify the data as appropriate.

Review initial calibration Form VIs and the associated laboratory raw data. Determine which compounds have average Relative Response Factors (RRFs) <0.050 and which compounds have Percent Relative Standard Deviations (%RSDs) >50 and between 30%-50%. Circle these noncompliances on your working copies of these Forms. Spot-check (i.e., recalculate) a few of the RRFs and %RSDs to verify the laboratory's computation.

Determine which samples are affected by reviewing the continuing calibration Form VIIs. Check the initial calibration date(s) noted in the headings of the Form VIIs to determine which continuing calibrations are associated with which initial calibrations. Next, review the sample listings given on the data package Form Vs. Match the indicated continuing calibration run with the appropriate Form VII by matching the laboratory file ID numbers. Write the affected samples (those listed on the matched Form V) on your working copies of the appropriate Form VI and VII.

Review the continuing calibration Form VIIs and the associated laboratory raw data. Determine which compounds have RRFs <0.050 and which compounds have Percent Differences (%Ds) >50, and between 25%-50%; circle the noncompliances on your working copies of these Forms. Spot-check (i.e., recalculate) a few of the RRFs and %Ds to verify the laboratory's computation.

Generally, affected positive results for compounds for which RRFs are <0.050 are qualified as estimated (J); nondetects are rejected (R). In accordance with some USEPA Regional protocol, the (L) qualifier may be used instead of (J) when qualifying positive results. Bias for these results is very low.

Generally, positive results for compounds for which %RSD exceeds 50% or %D exceeds 25% are qualified as estimated (J); nondetects (UJ). Check the specific applicable data validation protocol for further guidance as there are some protocols which reject nondetects if the %RSD or %D is excessive. Bias for these results cannot be determined.

Generally, positive results for compounds for which %RSD is between 30%-50% are qualified as estimated, (J). Qualification of nondetects is protocol-specific. Follow the rules given in the appropriate validation protocol.

1.1.1.6.3 Blank Contamination

When using the information given below and in the appropriate USEPA Regional Functional Guidelines, keep in mind that the validation action levels derived are sample specific and must be adjusted for dilution, sample aliquot used for analysis, and sample moisture content (when applicable).

The rules for qualifying data based on the occurrence of blank contamination vary based on regional protocols; The guidelines provided in the appropriate protocol should be followed.

Generally the blank contamination review process is completed by first considering the maximum amount of a particular contaminant occurring in the laboratory method blanks. (Do not consider lab blanks run after high concentration samples for purposes of determining carryover as laboratory method blanks!) Then repeat the process for contaminants occurring in the associated field quality control blanks. Action levels for qualification (10X or 5X depending upon whether or not the contaminant is a common contaminant) are then set. The list of common contaminants may vary among protocols. Additionally, some hierarchy among the field quality control blanks apply, and the manner in which the qualifiers are applied vary [i.e. use of (U) or (B); replacement by CRQL, etc.]. Refer to appropriate protocol for specific guidance.

1.1.1.6.4 Surrogates

Surrogates are evaluated by reviewing the laboratory data package Form II reports and the laboratory raw data. The quality control ranges are given on the laboratory data package Form IIs; circle any noncompliances on your working copies of these Forms.

Results for all compounds in an affected sample are qualified if any one of the surrogate spike compounds fail to meet the quality control criteria provided. Generally, for samples having a surrogate recovery <10%, positive results are qualified as estimated (J), nondetects are rejected (R). These results are biased low. For samples having a surrogate recovery which is low but >10%, positive results are generally qualified as estimated (J); nondetects (UJ). The bias qualifiers (L, UL) may be used instead, depending upon the specific USEPA Regional guidance. For samples having a surrogate recovery which is high, positive results are generally qualified as estimated (J, K) based on regional guidance; nondetects are not qualified based on high surrogate recoveries.

1.1.1.6.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

Generally, no data are qualified based upon MS/MSD results alone. If qualification does occur, generally only the result for that particular noncompliant compound is qualified in the original unspiked sample. Refer to the applicable data validation protocol for specific procedures for appropriately evaluating MS/MSD analyses.

1.1.1.6.6 Internal Standards

Internal standards are evaluated by reviewing the data package Form VIIIs and the laboratory raw data. The quality control ranges are given on the Form VIIIs. Circle any noncompliances on your working copies of these forms; evaluate and qualify as stipulated in the appropriate data validation protocol.

1.1.1.6.7 Tentatively Identified Compounds (TICs)

TICs are evaluated using the laboratory data package Form I VOA-TIC reports and the laboratory raw data. The guidance given in the March 1990 National Functional Guidelines for USEPA Region III is very concise; use the information in this document to evaluate and qualify accordingly.

1.1.1.6.8 Other Considerations

Laboratory precision can be evaluated by comparing the unspiked sample results with MS/MSD analyses results for unspiked compounds. Consider nondetects and results reported at concentrations less than the Contract Required Quantitation Limit (CRQL) to be in agreement. Use professional judgment in determining whether to qualify sample results based on the comparison.

Likewise, compare the positive compound results for field duplicate samples. Generally, the Relative Percent Difference (RPD) between field duplicate results for the aqueous matrix should be <35%; for soil matrix results, <50%. Qualification of the sample data is limited to the specific field duplicate pair. Positive results for compounds showing imprecision are qualified as estimated (J); nondetects (UJ). Bias for these results cannot be determined.

In some USEPA Regions, a "Percent Solids" rule applies. For example, if a sediment sample contains <50% solids in USEPA Region II, all associated data are considered to be estimated and are qualified accordingly. Follow the appropriate protocol guidance when applicable.

1.1.1.6.9 Quantitation

Verify and record the quantitation of at least one compound per analytical fraction. If no positive results are reported, use the MS/MSD data to confirm proper computation by the laboratory. Validator and laboratory quantitations must agree within 10 percent.

1.1.1.7 Deliverables Guidance

In addition to any specific USEPA Regional requirements (e.g. data validation memorandum, data summary spreadsheets, Regional worksheets), all laboratory data package quality control summary forms, sample Form I reports, method blank Form Is, and the Chain-of-Custody report must be given to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct (in accordance with the appropriate USEPA Regional or client requirements) and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

1.1.2 Semivolatiles (USEPA CLP Statement of Work (SOW) 3/90)

1.1.2.1 Applicability

CLP 3/90 semivolatile methodology is applicable to nearly all types of samples, regardless of water content, including groundwater, aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments. Method 8250 can be used to analyze groundwater samples as well.

The semivolatile TCL includes the following compounds:

Acenaphthene
Acenaphthylene
Anthracene
Benzo(a)anthracene
Benzo(b)fluoranthene
Benzo(k)fluoranthene
Benzo(g,h,i)perylene
Benzo(a)pyrene
4-Bromophenyl-phenylether
Butylbenzylphthalate
Carbazole
4-Chloroaniline
bis(2-Chloroethoxy)methane
bis(2-Chloroethyl)ether
4-Chloro-3-methylphenol
2-Chloronaphthalene
2-Chlorophenol
4-Chlorophenyl-phenylether
2,2'-oxybis(1-Chloropropane)
Chrysene
Dibenzo(a,h)anthracene
Dibenzofuran
1,2-Dichlorobenzene
1,3-Dichlorobenzene
1,4-Dichlorobenzene
3,3'-Dichlorobenzidine
2,4-Dichlorophenol
Diethylphthalate
2,4-Dimethylphenol
Dimethylphthalate
Di-n-butylphthalate
4,6-Dinitro-2-methylphenol
2,4-Dinitrophenol
2,4-Dinitrotoluene
2,6-Dinitrotoluene
Di-n-octylphthalate
bis(2-Ethylhexyl)phthalate
Fluoranthene
Fluorene
Hexachlorobenzene
Hexachlorobutadiene
Hexachlorocyclopentadiene
Hexachloroethane
Indeno(1,2,3-cd)pyrene
Isophorone
2-Methylnaphthalene
2-Methylphenol
4-Methylphenol
Naphthalene
2-Nitroaniline
3-Nitroaniline
4-Nitroaniline
Nitrobenzene
2-Nitrophenol
4-Nitrophenol
N-Nitroso-di-n-propylamine
N-Nitroso-diphenylamine
Pentachlorophenol
Phenanthrene
Phenol
Pyrene
1,2,4-Trichlorobenzene
2,4,5-Trichlorophenol
2,4,6-Trichlorophenol

The preceding method is based upon solvent extractions followed by gas chromatographic/mass spectrometric (GC/MS) procedures.

1.1.2.2 Interferences

Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated baselines causing misinterpretation of chromatograms. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by running method blanks. The use of high purity reagents and solvents helps to minimize interference problems; purification of solvents by distillation in all-glass systems may be required.

Interferences co-extracted from the samples will vary considerably from source to source depending upon the diversity of the industrial complex or waste being sampled.

1.1.2.3 General Laboratory Practices

An extraction blank should be prepared with each batch of samples extracted.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) analyses should be conducted to determine the effects of sample matrix upon the compounds of interest.

1.1.2.4 Sample Preparation

Prior to GC/MS analysis, aqueous samples are acidified to pH 2 and extracted with methylene chloride using a continuous liquid-liquid extractor. Both neat and diluted organic liquids may be analyzed by direct injection. Solid samples are extracted with 1:1 methylene chloride/acetone using a sonication procedure. Cleanup by Gel Permeation Chromatography (GPC) is required for solid sample extracts.

1.1.2.5 Data Overview to Validation

Before commencing validation, the reviewer must preview the associated Chain-of-Custody (COC) reports to determine:

- If the appropriate number of samples are present in the data package and if each sample was correctly analyzed for the parameters and methods specified.
- The identity of all associated field quality control blanks and field duplicate pairs.

Because many samples may have required dilutions, re-extraction and/or reanalyses, the data validator should preview the data package contents to determine which analyses represent the better quality data.

The data package should never be annotated unless specifically directed by client protocol. All Form I reports (including those for samples, laboratory method blanks, and MS/MSD analyses) and all laboratory quality control summary forms (including all initial and continuing calibration summary statistics) should be photocopied for use as working copies.

1.1.2.6 Technical Evaluation Summary

All data evaluations must be conducted in accordance with the appropriate USEPA Regional protocols (when applicable) and/or specified client contract requirements. The applicable documents must be referenced during the data validation process as this S.O.P. is only intended as a general procedure for all data validation tasks.

General parameters such as Data Completeness, Overall System Performance, Chromatographic Quality, Detection Limits and Compound Identification are evaluated concurrently with the parameters discussed in the following subsections.

1.1.2.6.1 Holding Times

Holding times are evaluated by reviewing the COC reports, the individual sample Form I reports, and the associated laboratory raw data. Holding times for extraction are calculated from date of collection to date of extraction.

The technical holding times for aqueous and solid matrices are as follows:

- Extraction: 7 days
- Analysis: 40 days from date of extraction

Affected positive results are generally qualified as estimated (J), nondetects (UJ). Alternately, the L or UL bias qualifiers may be used dependent upon the applicable USEPA Regional Guidance. If the sample was extracted beyond 14 days from collection, the holding time exceedance is considered to be gross and positive results are qualified as estimated (J) or (L); nondetects are rejected (R). Generally, if the holding time until extraction is exceeded, the affected sample results are considered to be biased low. If the holding time until analysis is exceeded (and potentially, some of the extract may have evaporated), the affected sample results may be considered to be biased high. Follow the qualification guidance given in the appropriate data validation protocol.

1.1.2.6.2 Calibration

Check that an initial calibration was performed for each instrument used for analysis and that all calibrations were performed at all appropriate concentration levels within 12 hours of the associated instrument tuning.

Review the data package Form Vs (tuning) using the applicable USEPA Regional Functional Guidelines, and qualify the data as appropriate.

Review initial calibration Form VIs and the associated laboratory raw data. Determine which compounds have average Relative Response Factors (RRFs) <0.050 and which compounds have Percent Relative Standard Deviations (%RSDs) >50 and between 30%-50%. Circle these noncompliances on your working copies of these Forms. Spot-check (i.e., recalculate) a few of the RRFs and %RSDs to verify the laboratory's computation.

Determine which samples are affected by reviewing the continuing calibration Form VIIs. Check the initial calibration date(s) noted in the headings of the Form VIIs to determine which continuing calibrations are associated with which initial calibrations. Next, review the sample listings given on the data package Form Vs. Match the indicated continuing calibration run with the appropriate Form VII by matching the laboratory file ID numbers. Write the affected samples (those listed on the matched Form V) on your working copies of the appropriate Form VI and VII.

Review the continuing calibration Form VIIs and the associated laboratory raw data. Determine which compounds have RRFs <0.050 and which compounds have Percent Differences (%Ds) >50, and between 25%-50%; circle the noncompliances on your working copies of these Forms. Spot-check (i.e., recalculate) a few of the RRFs and %Ds to verify the laboratory's computation.

Generally, affected positive results for compounds for which RRFs are <0.050 are qualified as estimated (J); nondetects are rejected (R). In accordance with some USEPA Regional protocol, the (L) qualifier may be used instead of (J) when qualifying positive results. Bias for these results is low.

Generally, positive results for compounds for which %RSD or %D exceeds 50% are qualified as estimated (J); nondetects (UJ). Check the specific applicable data validation protocol for further guidance as there are some protocol which reject nondetects if the %RSD or %D is excessive. Bias for these results cannot be determined.

Generally, positive results for compounds for which %RSD is between 30%-50% or %D is between 25%-50% are qualified as estimated (J). Qualification of nondetects is protocol-specific. Follow the rules given in the appropriate validation protocol.

1.1.2.6.3 Blank Contamination

Note that unlike VOA fraction analyses, a laboratory method blank does not have to be analyzed after every continuing calibration standard. Be very sure, however, that one semivolatile method blank was extracted for each day that associated samples were extracted (with a maximum of 20 samples per batch).

The action levels for qualification are 10X the maximum amount of phthalates found in the blanks (phthalates are common contaminants) and 5X the maximum amount of other contaminants found in the blanks. The actual action level applied is sample-specific and must be adjusted for dilution, sample aliquot used for analysis, and moisture content. The type and manner in which the qualifiers are applied

vary with protocol [i.e., use of (U) or (B); replacement by CRQL, etc.]. Refer to appropriate data validation protocol for specific guidance.

1.1.2.6.4 Surrogates

Surrogates are evaluated by reviewing the laboratory data package Form II reports and the associated laboratory raw data. The quality control ranges are given on the laboratory data package Form IIs; circle any noncompliances on your working copies of these Forms.

Semivolatile compounds are divided into two classes, base-neutral compounds and acid-extractable compounds. Each class of compounds has its own associated surrogates. If the recovery is <10% for any one surrogate, positive results for all compounds in that class in the affected sample are qualified as estimated, (J) or (L), and nondetects are rejected, (R). These results are biased low.

No qualification actions are taken for samples having any one surrogate recovery which is noncompliant but >10%.

If the recoveries for any two surrogates of the same class are noncompliant but above 10%, all sample results for that class of compounds in the affected sample are qualified. If the recoveries are low, positive results are generally qualified as estimated (J); nondetects (UJ). In some Regions, the bias qualifiers, L and UL, may be used instead. If the recoveries for any two surrogates of the same class are high, positive results for all compounds in that class in the affected sample are qualified, J or K, depending upon the appropriate USEPA Regional guidance; nondetects are not qualified based on high surrogate recoveries.

1.1.2.6.5 Matrix Spike/Matrix Spike Duplicates

Generally, no data are qualified based upon MS/MSD results alone. If qualification does occur, generally only the result for that particular noncompliant compound is qualified in the original unspiked sample. Refer to the appropriate validation guidelines for specific procedures for evaluating MS/MSD analyses.

1.1.2.6.6 Internal Standards

Internal standards are evaluated by reviewing the data package Form VIIIs and the laboratory raw data. The quality control ranges are given on the Form VIIIs. Circle any noncompliances on your working copies of these forms; evaluate and qualify as stipulated in the appropriate protocol.

1.1.2.6.7 Tentatively Identified Compounds (TICs)

TICs are evaluated using the laboratory data package Form I BNA-TIC reports, and the laboratory raw data. The guidance given in the 3/90 National Functional Guidelines for USEPA Region III is very concise; evaluate and qualify accordingly.

1.1.2.6.8 Other Considerations

Laboratory precision can be evaluated by comparing MS/MSD sample results for unspiked compounds with the unspiked sample results. Consider nondetects and results reported at concentration levels less than the Contract Required Quantitation Limit (CRQL) to be in agreement. Use professional judgment in determining whether to qualify sample results based on the comparison.

Likewise, compare the positive compound results for field duplicate samples. Generally the Relative Percent Difference (RPD) between field duplicate results for the aqueous matrix should be less than 35%; for soil matrix results, less than 50%. Qualification of sample data is limited to that specific field duplicate pair. Positive results for compounds showing imprecision are qualified as estimated (J); and nondetects (UJ). Bias for these results cannot be determined.

In some USEPA regions a "Percent Solids" rule applies. For example, if a sediment contains less than 50% solids in USEPA Region II, all associated data are considered to be estimated and are qualified accordingly. Follow the appropriate protocol guidance when applicable.

1.1.2.6.9 Quantitation

Verify and record quantitation of at least one compound per analytical fraction. If no positive results are reported, use the MS/MSD data to confirm proper computation by the laboratory. Validator and laboratory quantitations must agree within 10 percent.

1.1.2.7 Deliverables Guidance

In addition to any specific USEPA Regional requirements (e.g., data validation memorandum, data summary spreadsheets, USEPA Regional worksheets), all laboratory data package quality control summary forms, sample Form I reports, method blank Form Is, and the Chain-of-Custody report must be given to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct (in accordance with the appropriate USEPA Regional or client requirements) and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

1.2 CLP Organics by GC

1.2.1 Organochlorine Pesticides and Polychlorinated Biphenyls (PCBs) (USEPA CLP Statement of Work (SOW) 3/90)

1.2.1.1 Applicability

CLP 3/90 methodology is used to determine the concentration of certain organochlorine pesticides and polychlorinated biphenyls (PCBs) in groundwater, liquid, and solid sample matrices. Specifically, the CLP 3/90 TCL includes the following substances:

Aldrin	Dieldrin	Methoxychlor
alpha-BHC	Endosulfan I	Toxaphene
beta-BHC	Endosulfan II	Aroclor-1016
delta-BHC	Endosulfan sulfate	Aroclor-1221
gamma-BHC (Lindane)	Endrin	Aroclor-1232
Chlordane	Endrin aldehyde	Aroclor-1242
4,4'-DDD	Endrin ketone	Aroclor-1248
4,4'-DDE	Heptachlor	Aroclor-1254
4,4'-DDT	Heptachlor epoxide	Aroclor-1260

CLP 3/90 methodology for organochlorine pesticides and PCBs is a Gas Chromatographic (GC) procedure in which samples are first extracted and then analyzed by direct injection. The compounds

of interest are analyzed via GC/ECD (Electron Capture Detector; an equivalent Halogen-Specific Detector may also be used).

1.2.1.2 Interferences

The sensitivity of these methods usually depends on the level of interferences rather than on instrumental limitations. Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated baselines causing misinterpretation of gas chromatograms. The use of high purity reagents and solvents helps to minimize these interference problems. Extraction blanks are analyzed as method blanks in order to monitor the occurrences of interferences.

Interferences co-extracted from the sample will vary considerably and will dictate the nature and extent of clean-up procedures used. Phthalate esters are a common interference to organochlorine pesticide analyses; phenols and organic acids may act as interferents when analyzing for chlorinated herbicides.

1.2.1.3 General Laboratory Practices

An extraction blank should be prepared with each batch of samples extracted.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) analyses should be conducted to determine the effects of sample matrix upon the compounds of interest.

Standard quality assurance practices such as the analyses of field replicates should also be employed.

1.2.1.4 Sample Preparation

Prior to GC analysis, aqueous samples are extracted at a neutral pH with methylene chloride as a solvent using a separatory funnel or a continuous liquid-liquid extractor. Solid samples are extracted with hexane:acetone (1:1) using sonication procedures.

1.2.1.5 Data Overview Prior to Validation

Before commencing validation, the reviewer must preview the associated Chain-of-Custody (COC) reports to determine:

- If the appropriate number of samples are present in the data package and if each sample was correctly analyzed for the parameters and methods specified.
- The identity of all associated field quality control blanks and field duplicate pairs.

Because many samples may have required dilutions, re-extractions and/or re-analyses, the validator should preview the data package contents to determine which analyses represent the better quality data.

Unless specifically directed by client protocol, never annotate the laboratory data package. Before beginning evaluation, prepare working copies (i.e., photocopies) of all Form I reports (including those for samples, laboratory method blanks and MS/MSD analyses) and all laboratory quality control summary forms.

1.2.1.6 Technical Evaluation Summary

All data evaluations must be conducted in accordance with applicable USEPA Regional protocols (when applicable) and/or specific client contract requirements. The applicable documents must be referenced during the data evaluation process as this S.O.P. is only intended as a general procedure for the data validation tasks.

General parameters such as Data Completeness, Overall System Performance, Chromatographic Quality, Detection Limits, and Compound Identification are evaluated concurrently with the parameters discussed in the following subsections.

1.2.1.6.1 Holding Times

Holding times are evaluated by reviewing the COC reports, the individual sample Form I reports, and the associated laboratory raw data. Holding times are calculated from date of collection to date of extraction/analysis.

All samples to be analyzed for pesticides, PCBs and/or herbicides must be extracted within 7 days of collection regardless of matrix and analyzed within 40 days of extraction.

When the holding time criteria are not met, positive results in affected samples are generally qualified as estimated (J); nondetects (UJ). These results are biased low. Some USEPA Regions apply the bias qualifiers, L and UL, instead. If the holding times are exceeded by a factor of 2 or more, the holding time exceedance is considered to be gross, and positive results are generally qualified as estimated (J); nondetects are generally considered to be unreliable and are rejected (R). These results are biased low.

1.2.1.6.2 Calibration

Data pertaining to the initial calibration (i.e., evaluation check for linearity) is found on the data package Form VIs. Check that the initial calibration was performed for each instrument used and at all appropriate concentration levels.

Generally, positive results for compounds whose Percent Relative Standard Deviation (%RSD) exceeds 20% are qualified as estimated (J). Check the pesticide analytical sequence (Form VIII) to determine which samples are affected. Check the specific applicable data validation protocol for further guidance as there are some protocol which reject nondetects if the %RSD or %D is excessive. Bias for these results cannot be determined. Follow the rules given in the appropriate data validation protocol.

Verify that a resolution check mixture, Performance Evaluation Mixture (PEM), Individual Standard Mixtures A and B, and multicomponent target compounds were analyzed at the proper frequency (see Form VIII) on each GC analytical column. Retention times for PEM target compounds and Individual Standard Mixtures A and B target compounds should be within the established retention time windows. If a compound is outside of the retention time window, further evaluation of the sample chromatograms is necessary. In addition, check that the Relative Percent Difference (RPD) (recorded on Forms VII-D and VII-E) between the calculated amount and true amount for each pesticide is $\leq 25\%$. If this criterion is not met, positive results and nondetects for the affected compounds are qualified as estimated, (J) and (UJ), respectively.

The DDT/Endrin Breakdown for each PEM should not exceed 20% (recorded on Form VII-D). Generally, if % breakdown for DDT exceeds 20%, estimate (J) all positive results for DDT, DDE and DDD following the last in-control standard until the next acceptable PEM (see analytical sequence); acceptability of the

next individual A/B mix may also be considered when qualifying data for DDT breakdown. If there are no positive results for DDT but there are positive results for DDD or DDE then reject (R) nondetects for DDT in associated samples. Generally, if Endrin % Breakdown exceeds 20%, estimate (J) positive results for Endrin, Endrin Aldehyde, and Endrin Ketone in all samples following the last in-control standard until the next acceptable PEM; acceptability of the next individual A/B mix may also be considered when qualifying data for Endrin breakdown. If there are positive results for Endrin Aldehyde or Endrin Ketone but none for Endrin, reject (R) nondetect Endrin results.

1.2.1.6.3 Blank Contamination

When using the information provided below and in the appropriate data validation guidelines, keep in mind that the validation action levels derived are sample-specific and must be adjusted for dilution, sample aliquot used for analysis, and sample moisture content (when applicable).

The rules for qualifying data based on the occurrence of blank contamination vary based on regional protocols; Guidelines provided in the appropriate data validation protocol should be followed.

An action level of 5X the maximum amount of contaminant found is used to evaluate the sample data. The manner in which the qualifiers are applied vary [i.e. use of (U) or (B); replacement by CRQL, etc.]. Refer to appropriate validation protocol for specific guidance.

1.2.1.6.4 Surrogates

Surrogates are evaluated by reviewing the laboratory data package Form II reports and the associated laboratory raw data. The advisory limits are given on the laboratory data package Form IIs; circle any recoveries outside these limits on your working copies of these Forms.

No qualifications are made for surrogates which show zero recoveries because they were "diluted out." Generally, positive results affected by low surrogate recovery are qualified as estimated (J), or the (L) bias qualifier is used when applicable; nondetects are qualified (UJ) or (UL), accordingly. If a positive sample result is affected by high surrogate recovery, the result is qualified as estimated (J) or the (K) bias qualifier is used when applicable; nondetects are not qualified based on high surrogate recovery. Because the surrogate recovery limits for this fraction are advisory, generally no results are rejected.

The decachlorobiphenyl (DCB) and tetrachloro-m-xylene (TCX) retention times found on data package Form VIII must be ±0.10 for DCB and ±0.05 for TCX. If DCB and TCX retention time criteria are not met, the raw data must be checked for misidentified GC peaks. The validator's professional judgment for qualifications should be used.

1.2.1.6.5 Matrix Spike/Matrix Spike Duplicates

Generally, no data are qualified based upon MS/MSD results alone. If qualification does occur, generally only the result for that particular noncompliant compound is qualified in the original unspiked sample. Refer to the appropriate data validation guidelines for the specific procedures for evaluating MS/MSD analyses.

1.2.1.6.6 Other Considerations

Laboratory precision can be evaluated by comparing the unspiked sample results with MS/MSD analyses results for unspiked compounds. Consider nondetect results and results reported at concentrations less

than the Contract Required Quantitation Limit (CRQL) to be in agreement. Use professional judgment in determining whether to qualify sample results based on the comparison.

Likewise, compare the positive compound results for field duplicate samples. Generally, the Relative Percent Difference (RPD) between field duplicate results for the aqueous matrix should be <35%; for soil matrix results, <50%. Qualification of the sample data is limited to the specific field duplicate pair. Positive results for compounds showing imprecision are qualified as estimated (J); nondetects (UJ). Bias for these results cannot be determined.

In some USEPA Regions, a "Percent Solids" rule applies. For example, if a sediment sample contains <50% solids in USEPA Region II, all associated data are considered to be estimated, and are qualified accordingly. Follow the appropriate protocol guidance when applicable.

1.2.1.6.7 Quantitation

Verify and record the quantitation of at least one compound per analytical fraction. If no positive results are reported, use the MS/MSD data to confirm proper computation by the laboratory. Validator and laboratory quantitations must agree within 10%.

1.2.1.7 Deliverables Guidance

In addition to any specific USEPA Regional requirements (e.g., data validation memorandum, data summary spreadsheets, USEPA Regional worksheets), all laboratory data package quality control summary forms, sample Form I reports, method blank Form Is, and the Chain-of-Custody report must be given to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct (in accordance with the appropriate USEPA Regional or client requirements) and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

2.0 NON-CLP ORGANICS FOR SOLID MATRICES

2.1 SW-846 Organics by GC/MS

2.1.1 Volatiles (Method 8240B, 8260A)

2.1.1.1 Applicability

Method 8240 is used to determine volatile organic compounds in most waste matrices including groundwater, sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments.

Method 8240 analyte list includes of the volatile CLP 3/90 Target Compound List (TCL) (Section 1.1.1) plus the following compounds*:

Acetonitrile	trans-1,2-Dichloroethene
Acrolein	Ethyl methacrylate
Acrylonitrile	Iodomethane
Allyl chloride	Methacrylonitrile
Chloropropene	Methyl methacrylate
1,2-Dibromo-3-chloropropane	2-Picoline
1,2-Dibromoethane	Pyridine
Dibromomethane	Trichlorofluoromethane
trans-1,4-Dichloro-2-butene	1,2,3-Trichloropropane
Dichlorodifluoromethane	Vinyl acetate

* Appendix IX target compounds

Method 8240 is based upon a purge-and-trap, gas chromatographic/mass spectrometric (GC/MS) procedure. Prior to analysis, samples must be prepared by Method 5030.

2.1.1.2 Interferences

Samples can be contaminated by diffusion of volatile organics (particularly chlorofluorocarbons and methylene chloride) through the sample container septum during shipment and storage. Associated field quality control blanks are analyzed in order to monitor this.

Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe or purging device is rinsed out between samples with reagent water. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of reagent water to check for cross contamination.

If sample or matrix interferences are encountered, a secondary or alternate analytical column may be used to resolve the compounds of interest.

2.1.1.3 General Laboratory Practices

A method blank consisting of organic free water spiked with surrogates and internal standards should be analyzed immediately following each daily calibration and also after the analysis of every high concentration sample.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) analyses should be conducted to determine the effects of sample matrix upon the compounds of interest.

2.1.1.4 Sample Preparation

Method 5030 is a purge-and-trap procedure performed to prepare and extract volatile compounds from samples and introduce those compounds into the GC/MS.

For highly volatile matrices, direct injection preceded by dilution should be used to prevent gross contamination of the instrumentation. For pastes, dilution of the sample until it becomes free-flowing is used to ensure adequate interfacial area. The success of this method depends on the level of interferences in the sample; results may vary due to the large variability and complicated matrices of solid waste samples.

2.1.1.5 Data Overview Prior to Validation

Before commencing validation, the reviewer must preview the associated Chain-of-Custody (COC) reports to determine:

- If the appropriate number of samples are present in the data package and if each sample was correctly analyzed for the parameters and methods specified.
- The identity of all associated field quality control blanks and field duplicate pairs.

Because many samples may have required dilutions, re-extractions and/or re-analyses, the validator should preview the data package contents to determine which analyses represent the better quality data.

Unless specifically directed by client protocol, never annotate the laboratory data package. Before beginning evaluation, prepare working copies (i.e., photocopies) of all Form I reports (including those for samples, laboratory method blanks and MS/MSD analyses) and all laboratory quality control summary forms (including all initial and continuing calibration summary statistics).

2.1.1.6 Technical Evaluation Summary

All data evaluations must be conducted in accordance with applicable USEPA Regional protocols and/or specific client contract requirements. The applicable documents must be referenced during the data evaluation process as this S.O.P. is only intended as a general procedure for the data validation tasks.

General parameters such as Data Completeness, Overall System Performance, Chromatographic Quality, Detection Limits and Compound Identification are evaluated concurrently with the parameters discussed in the following subsections.

2.1.1.6.1 Holding Times

Holding times are evaluated by reviewing the COC reports, the individual sample Form I reports, and the associated laboratory raw data. Holding times are calculated from date of collection to date of analysis.

The technical maximum holding time allowance for aqueous samples preserved with hydrochloric acid (HCL) is 14 days.

No technical holding times for solid matrices have been promulgated: a 14-day maximum holding time allowance is currently being used.

For unpreserved aqueous samples, generally a 7-day maximum holding time allowance for aromatic compounds, along with a 14-day maximum holding time allowance for chlorinated hydrocarbons is used.

Positive results in affected samples are generally qualified as estimated (J); nondetects (UJ). These results are biased low. Some USEPA Regions apply the bias qualifiers, L and UL, instead. If the holding times are exceeded by a factor of 2 or more, the holding time exceedance is considered to be gross and positive results are generally qualified as estimated (J): nondetects are generally considered to be unreliable and are qualified (R). Results for which the holding time was grossly exceeded are biased low.

2.1.1.6.2 Calibration

Check that an initial calibration was performed for each instrument used for analysis and that all calibrations were performed at all appropriate concentration levels within 12 hours of the associated instrument tuning.

Review the data package Form Vs (tuning) using the applicable USEPA Regional Functional Guidelines, and qualify the data as appropriate.

Review initial calibration Form VIs and the associated laboratory raw data. Determine which compounds have average Relative Response Factors (RRFs) <0.050 and which compounds have Percent Relative Standard Deviations (%RSDs) >50% and between 30% and 50%. Circle these noncompliances on your working copies of these Forms. Spot-check (i.e., recalculate) a few of the RRFs and %RSDs to verify the laboratory's computation.

Determine which samples are affected by reviewing the continuing calibration Form VIIs. Check the initial calibration date(s) noted in the headings of the Form VIIs to determine which continuing calibrations are associated with which initial calibrations. Next, review the sample listings given on the data package Form Vs. Match the indicated continuing calibration run with the appropriate Form VII by matching the laboratory file ID numbers. Write the affected samples (those listed on the matched Form V) on your working copies of the appropriate Form VI and VII. Spot-check (i.e., recalculate) a few of the RRFs and %Ds to verify the laboratory's computation.

Review the continuing calibration Form VIIs and the associated laboratory raw data. Determine which compounds have RRFs <0.050 and which compounds have Percent Differences (%Ds) >25%; circle the noncompliances on your working copies of these Forms.

Generally, affected positive results for compounds whose RRFs are <0.050 are qualified as estimated (J); nondetects are rejected (R). In accordance with some USEPA Regional protocol, the (L) qualifier may be used instead of (J), when qualifying positive results. Bias for these results is low.

Generally, positive results for compounds for which %RSD exceeds 50% or %D exceeds 25% are qualified as estimated (J); nondetects (UJ). Check the specific applicable data validation protocol for further guidance as there are some protocol which reject nondetects if the %RSD or %D is excessive. Bias for these results cannot be determined.

Generally, positive results for compounds for which %RSD is between 30%-50% are qualified as estimated (J). Qualification of nondetects is protocol-specific. Follow the rules given in the appropriate validation protocol.

2.1.1.6.3 Blank Contamination

When using the information given below and in the appropriate USEPA Regional Functional Guidelines, keep in mind that the validation action levels derived are sample-specific and must be adjusted for dilution, sample aliquot used for analysis, and sample moisture content (when applicable).

The rules for qualifying data based on the occurrence of blank contamination vary based on regional protocols. The guidelines provided in the appropriate protocol should be followed.

Generally the blank contamination review process is completed by first considering the maximum amount of a particular contaminant occurring in the laboratory method blanks. (Do not consider lab blanks run after high concentration samples for purposes of determining carryover as laboratory method blanks!) Then repeat the process for contaminants occurring in the associated field quality control blanks. Action levels for qualification (10X or 5X depending upon whether or not the contaminant is a common contaminant) are then set. The list of common contaminants may vary among protocols. Additionally, some hierarchy among the field quality control blanks apply, and the manner in which the qualifiers are applied vary [i.e. use of (U) or (B); replacement by CRQL, etc.]. Refer to appropriate protocol for specific guidance.

2.1.1.6.4 Surrogates

Surrogates are evaluated by reviewing the laboratory data package Form II reports and the laboratory raw data. The quality control ranges are given on the laboratory data package Form IIs; circle any noncompliances on your working copies of these Forms.

Results for all compounds in an affected sample are qualified if any one of the surrogate spike compounds fail to meet the quality control criteria provided. Generally, for samples having a surrogate recovery <10%, positive results are qualified as estimated (J), nondetects are rejected (R). These results are biased low. For samples having a surrogate recovery which is low but >10%, positive results are generally qualified as estimated (J); nondetects (UJ). The bias qualifiers (L, UL) may be used instead, depending upon the specific USEPA Regional guidance. For samples having a surrogate recovery which is high, positive results are generally qualified as estimated (J, K) based on regional guidance, nondetects are not qualified based on high surrogate recovery.

2.1.1.6.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

Generally, no data are qualified based upon MS/MSD results alone. If qualification does occur, generally only the result for that particular noncompliant compound is qualified in the original unspiked sample. Refer to the applicable data validation protocol for specific procedures for appropriately evaluating MS/MSD analyses.

2.1.1.6.6 Internal Standards

Internal standards are evaluated by reviewing the data package Form VIIIs and the laboratory raw data. The quality control ranges are given on the Form VIIIs. Circle any noncompliances on your working copies of these forms; evaluate and qualify as stipulated in the appropriate data validation protocol.

2.1.1.6.7 Tentatively Identified Compounds (TICs)

TICs are evaluated using the laboratory data package Form I VOA-TIC reports and the laboratory raw data. The guidance given in the March 1990 National Functional Guidelines for USEPA Region III is very concise; use the information in this document to evaluate and qualify accordingly.

2.1.1.6.8 Other Considerations

Laboratory precision can be evaluated by comparing the unspiked sample results with MS/MSD analyses results for unspiked compounds. Consider nondetects and results reported at concentrations less than the Contract Required Quantitation Limit (CRQL) to be in agreement. Use professional judgment in determining whether to qualify sample results based on the comparison.

Likewise, compare the positive compound results for field duplicate samples. Generally, the Relative Percent Difference (RPD) between field duplicate results for the aqueous matrix should be <35%; for soil matrix results, <50%. Qualification of the sample data is limited to the specific field duplicate pair. Positive results for compounds showing imprecision are qualified as estimated (J); nondetects (UJ). Bias for these results cannot be determined.

In some USEPA Regions, a "Percent Solids" rule applies. For example, if a sediment sample contains <50% solids in USEPA Region II, all associated data are considered to be estimated and are qualified accordingly. Follow the appropriate protocol guidance when applicable.

2.1.1.6.9 Quantitation

Verify and record the quantitation of at least one compound per analytical fraction. If no positive results are reported, use the MS/MSD data to confirm proper computation by the laboratory. Validator and laboratory quantitations must agree within 10 percent.

2.1.1.7 Deliverables Guidance

In addition to any specific USEPA Regional requirements (e.g. data validation memorandum, data summary spreadsheets, Regional worksheets), all laboratory data package quality control summary forms, sample Form I reports, method blank Form Is, and the Chain-of-Custody report must be given to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct (in accordance with the appropriate USEPA Regional or client requirements) and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

2.1.2 Semivolatiles (Method SW8250A, 8270B)

2.1.2.1 Applicability

Methods 8250 and 8270 are applicable to most types of samples, regardless of water content, including groundwater, aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments. Method 8250 can be used to analyze groundwater samples as well.

These methods can be used to quantify most neutral, acidic, and basic organic compounds that are soluble in methylene chloride and capable of elution without derivatization as sharp peaks from a gas

chromatographic column. Such compounds include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols, including nitrophenols.

The above methods specifically analyze for the semivolatile CLP 3/90 Target Compound List (TCL) (Section 1.1.2) plus the following compounds*:

Acetophenone
Aniline
Benzyl alcohol
Bis(2-chloroisopropyl)ether
Chlorobenzilate
Diallate
2,6-Dichlorophenol
Dimethoate
p-Dimethylaminoazobenzene
7,12-Dimethylbenz(a)anthracene
3,3'-Dimethylbenzidine
a,a-Dimethylphenylamine
1,3-Dinitrobenzene
Diphenylamine
Ethyl methanesulfonate
Famphur
Hexachlorophene
Hexachloropropene
Isodrin
Isosafrole
Kepone
Methapyrilene
3-Methylcholanthrene
Methyl methanesulfonate
3-Methylphenol
1,4-Naphthoquinone
4-Nitroquinoline-1-oxide
1-Naphthylamine
2-Naphthylamine
5-Nitro-o-toluidine
N-nitrosodiethylamine
N-nitrosodimethylamine
N-nitrosodimethylethylamine
N-nitroso-di-n-butylamine
N-nitrosomorpholine
N-nitrosopiperidine
Pentachlorobenzene
Pentachloronitrobenzene
Phenacetin
p-Phenylenediamine
Phorate
2-Picoline
Pronamide
Safrole
1,2,4,5-Tetrachlorobenzene
Thionazin
o,o,o-Triethylphosphorothioate
1,3,5-Trinitrobenzene

* Appendix IX target compounds

The preceding methods are based upon solvent extractions followed by gas chromatographic/mass spectrometric (GC/MS) procedures, Method 8250 being a GC/MS method using the packed column technique, and Method 8270 using GC/MS capillary column technique.

2.1.2.2 Interferences

Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated baselines causing misinterpretation of chromatograms. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by running method blanks. The use of high purity reagents and solvents helps to minimize interference problems; purification of solvents by distillation in all-glass systems may be required.

Interferences co-extracted from the samples will vary considerably from source to source, depending upon the diversity of the industrial complex or waste being sampled.

2.1.2.3 General Laboratory Practices

An extraction blank should be prepared with each batch of samples extracted.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) analyses should be conducted to determine the effects of sample matrix upon the compounds of interest.

2.1.2.4 Sample Preparation

Prior to analysis, the samples must be extracted using the appropriate techniques. Aqueous samples are extracted at the appropriate pH with methylene chloride as a solvent using a separatory funnel (Method 3510) or a continuous liquid-liquid extractor (Method 3520). Both neat and diluted organic liquids may be analyzed by direct injection. Solid samples are extracted at the appropriate pH with methylene chloride using either Soxhlet Extraction (Method 3540) or sonication (Method 3550) procedures.

2.1.2.5 Data Overview to Validation

Before commencing validation, the reviewer must preview the associated Chain-of-Custody (COC) reports to determine:

- If the appropriate number of samples are present in the data package and if each sample was correctly analyzed for the parameters and methods specified.
- The identity of all associated field quality control blanks and field duplicate pairs.

Because many samples may have required dilutions, re-extraction and/or reanalyses, the data validator should preview the data package contents to determine which analyses represent the better quality data.

The data package should never be annotated unless specifically directed by client protocol. All Form I reports (including those for samples, laboratory method blanks, and MS/MSD analyses) and all laboratory quality control summary forms (including all initial and continuing calibration summary statistics) should be photocopied for use as working copies.

2.1.2.6 Technical Evaluation Summary

All data evaluations must be conducted in accordance with the appropriate USEPA Regional protocols and/or specified client contract requirements. The applicable documents must be referenced during the data validation process as this S.O.P. is only intended as a general procedure for all data validation tasks.

General parameters such as Data Completeness, Overall System Performance, Chromatographic Quality, Detection Limits and Compound Identification are evaluated concurrently with the parameters discussed in the following subsections.

2.1.2.6.1 Holding Times

Holding times are evaluated by reviewing the COC reports, the individual sample Form I reports, and the associated laboratory raw data. Holding times for extraction are calculated from date of collection to date of extraction.

The technical holding times for aqueous and solid matrices are as follows:

- Extraction:
  - Water samples: 7 days
  - Solid samples: 14 days
- Analysis: 40 days from date of extraction

Affected positive results are generally qualified as estimated (J), nondetects (UJ). Alternately, the L or UL bias qualifiers may be used dependent upon the applicable USEPA Regional Guidance. If the sample was extracted beyond 14 days from collection (28 days for solid samples), the holding time exceedance is considered to be gross and positive results are qualified as estimated (J) or (L); nondetects are rejected (R). Generally, if the holding time until extraction is exceeded, the affected sample results are considered to be biased low. If the holding time until analysis has been exceeded (and potentially, some of the extract may have evaporated), the affected sample results may be considered to be biased high. Follow the qualification guidance given in the appropriate data validation protocol.

2.1.2.6.2 Calibration

Check that an initial calibration was performed for each instrument used for analysis and that all calibrations were performed at all appropriate concentration levels within 12 hours of the associated instrument tuning.

Review the data package Form Vs (tuning) using the applicable USEPA Regional Functional Guidelines, and qualify the data as appropriate.

Review initial calibration Form VIs and the associated laboratory raw data. Determine which compounds have average Relative Response Factors (RRFs) <0.050 and which compounds have Percent Relative Standard Deviations (%RSDs) >50% and between 30% and 50%. Circle these noncompliances on your working copies of these Forms. Spot-check (i.e., recalculate) a few of the RRFs and %RSDs to verify the laboratory's computation.

Determine which samples are affected by reviewing the continuing calibration Form VIIs. Check the initial calibration date(s) noted in the headings of the Form VIIs to determine which continuing calibrations are associated with which initial calibrations. Next, review the sample listings given on the data package Form Vs. Match the indicated continuing calibration run with the appropriate Form VII by matching the laboratory file ID numbers. Write the affected samples (those listed on the matched Form V) on your working copies of the appropriate Form VI and VII. Spot-check (i.e., recalculate) a few of the RRFs and %Ds to verify the laboratory's computation.

Review the continuing calibration Form VIIs, and the associated laboratory raw data. Determine which compounds have RRFs <0.050 and which compounds have Percent Differences (%Ds) >30%; circle the noncompliances on your working copies of these Forms.

Generally, affected positive results for compounds for which RRFs are <0.050 are qualified as estimated (J); nondetects are rejected (R). In accordance with some USEPA Regional protocol, the (L) qualifier may be used instead of (J) when qualifying positive results. Bias for these results is low.

Generally, positive results for compounds for which %RSD exceeds 50% or %D exceeds 30%, are qualified as estimated (J); nondetects (UJ). Check the specific applicable data validation protocol for further guidance as there are some protocol which reject nondetects if the %RSD or %D is excessive. Bias for these results cannot be determined.

Generally, positive results for compounds for which %RSD is between 30%-50% are qualified as estimated (J). Qualification of nondetects is protocol-specific. Follow the rules given in the appropriate validation protocol.

2.1.2.6.3 Blank Contamination

Note that unlike VOA fraction analyses, a laboratory method blank does not have to be analyzed after every continuing calibration standard. Be very sure, however, that one semivolatile method blank was extracted for each day that associated samples were extracted (with a maximum of 20 samples per batch).

The action levels for qualification are 10X the maximum amount of phthalates found in the blanks (phthalates are common contaminants) and 5X the maximum amount of other contaminants found in the blanks. The actual action level applied is sample-specific and must be adjusted for dilution, sample aliquot used for analysis, and moisture content. The type and manner in which the qualifiers are applied vary with protocol [i.e., use of (U) or (B); replacement by CRQL, etc.]. Refer to appropriate data validation protocol for specific guidance.

2.1.2.6.4 Surrogates

Surrogates are evaluated by reviewing the laboratory data package Form II reports and the associated laboratory raw data. The quality control ranges are given on the laboratory data package Form IIs; circle any noncompliances on your working copies of these Forms.

Semivolatile compounds are divided into two classes, base-neutral compounds and acid-extractable compounds. Each class of compounds has its own associated surrogates. If the recovery is <10% for any one surrogate, positive results for all compounds in that class in the affected sample are qualified as estimated, (J) or (L), and nondetects are rejected, (R). These results are biased low.

No qualification actions are taken for samples having any one surrogate recovery which is noncompliant but >10%.

If the recoveries for any two surrogates of the same class are noncompliant but above 10%, all sample results for that class of compounds in the affected sample are qualified. If the recoveries are low, positive results are generally qualified as estimated (J); nondetects (UJ). In some Regions, the bias qualifiers, L and UL, may be used instead. If the recoveries for any two surrogates of the same class are high, positive results for all compounds in that class in the affected sample are qualified, J or K, depending upon the appropriate USEPA Regional guidance; nondetects are not qualified based on high surrogate recoveries.

2.1.2.6.5 Matrix Spike/Matrix Spike Duplicates

Generally, no data are qualified based upon MS/MSD results alone. If qualification does occur, generally only the result for that particular noncompliant compound is qualified in the original unspiked sample analysis. Refer to the appropriate validation guidelines for specific procedures for evaluating MS/MSD analyses.

2.1.2.6.6 Internal Standards

Internal standards are evaluated by reviewing the data package Form VIIIs and the laboratory raw data. The quality control ranges are given on the Form VIIIs. Circle any noncompliances on your working copies of these forms; evaluate and qualify as stipulated in the appropriate protocol.

2.1.2.6.7 Tentatively Identified Compounds (TICs)

TICs are evaluated using the laboratory data package Form I BNA-TIC reports and the laboratory raw data. The guidance given in the 3/90 National Functional Guidelines for USEPA Region III is very concise; evaluate and qualify accordingly.

2.1.2.6.8 Other Considerations

Laboratory precision can be evaluated by comparing MS/MSD sample results for unspiked compounds with the unspiked sample results. Consider nondetects and results reported at concentration levels less than the Contract Required Quantitation Limit (CRQL) to be in agreement. Use professional judgment in determining whether to qualify sample results based on the comparison.

Likewise, compare the positive compound results for field duplicate samples. Generally the Relative Percent Difference (RPD) between field duplicate results for the aqueous matrix should be less than 35%; for soil matrix results, less than 50%. Qualification of sample data is limited to that specific field duplicate pair. Positive results for compounds showing imprecision are qualified as estimated (J); and nondetects (UJ). Bias for these results cannot be determined.

In some USEPA regions a "Percent Solids" rule applies. For example, if a sediment contains less than 50% solids in USEPA Region II, all associated data are considered to be estimated and are qualified accordingly. Follow the appropriate protocol guidance when applicable.

2.1.2.6.9 Quantitation

Verify and record quantitation of at least one compound per analytical fraction. If no positive results are reported, use the MS/MSD data to confirm proper computation by the laboratory. Validator and laboratory quantitations must agree within 10 percent.

2.1.2.7 Deliverables Guidance

In addition to any specific USEPA Regional requirements (e.g., data validation memorandum, data summary spreadsheets, USEPA Regional worksheets), all laboratory data package quality control summary forms, sample Form I reports, method blank Form Is, and the Chain-of-Custody report, must be given to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct (in accordance with the appropriate USEPA Regional or client requirements) and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

2.2 SW846 Non-CLP Organics by Gas Chromatography

2.2.1 Volatiles (SW 5030/SW 8010B, 8015A, 8020A, 8030A)

2.2.1.1 Applicability

Method 8010B is used to determine the concentration of the following halogenated volatile organic compounds in groundwater, liquid, and solid matrices:

Allyl chloride
Benzyl chloride

Bis (2-chloroethoxy)methane
Bis (2-chloroisopropyl)ether
Bromoacetone
Bromobenzene
Bromodichloromethane
Bromoform
Bromomethane
Carbon tetrachloride
Chlorobenzene
Chloroethane
2-Chloroethanol
Chloroform
1-Chlorohexane
2-Chloroethyl vinyl ether
Chloromethane
Chloromethyl methyl ether
Chloroprene
4-Chlorotoluene
Dibromochloromethane
1,2-Dibromo-3-chloropropane
Dibromomethane
1,2-Dichlorobenzene
1,3-Dichlorobenzene
1,4-Dichlorobenzene
Dichlorodifluoromethane
1,1-Dichloroethane
1,2-Dichloroethane
1,1-Dichloroethylene (Vinylidene chloride)
trans-1,2-Dichloroethylene
Dichloromethane
1,2-Dichloropropane
1,3-Dichloro-2-propanol
cis-1,3-Dichloropropene
trans-1,3-Dichloropropene
Epichlorhydrin
Ethylene dibromide
Methyl iodide
1,1,2,2-Tetrachloroethane
1,1,1,2-Tetrachloroethane
Tetrachloroethylene
1,1,1-Trichloroethane
1,1,2-Trichloroethane
Trichloroethylene
Trichlorofluoromethane
1,2,3-Trichloropropane
Vinyl chloride

Method 8015A is used to determine the concentration of the following nonhalogenated volatile organic compounds in groundwater, liquid, and solid matrices:

Diethyl ether
Ethanol
Methyl ethyl ketone (MEK)
Methyl isobutyl ketone (MIBK)

Method 8020A is used to determine the concentration of the following aromatic volatile organic compounds in groundwater, liquid, and solid matrices:

Benzene
Chlorobenzene
1,2-Dichlorobenzene
1,3-Dichlorobenzene
1,4-Dichlorobenzene
Toluene
Ethyl benzene
Xylenes (Dimethyl benzenes)

Method 8030A is used to determine the concentration of the following volatile organic compounds in groundwater, liquid, and solid matrices:

Acrolein (Propenal)
Acrylonitrile

All of the above Methods are gas chromatographic (GC) only (i.e., no mass spectrometer detector is employed). Method 8010B analyzes for halogenated volatile organics via GC/HSD (Halide-Specific Detector), Method 8015A analyzes for nonhalogenated volatile organics via GC/FID (Flame Ionization Detector), Method 8020A analyzes for aromatic Volatile organics via GC/PID (Photo-ionization Detector), and Method 8030A analyzes for the compounds acrolein and acrylonitrile using GC/FID. Samples can be analyzed by these methods using direct injection, the headspace method (Method 5020) or the purge-and-trap method (Method 5030). Groundwater samples should be determined using Method 5030.

2.2.1.2 Interferences

Samples can be contaminated by diffusion of volatile organics (particularly chlorofluorocarbons and methylene chloride) through the sample container septum during shipment and storage. Associated field quality control blanks are analyzed in order to monitor this.

Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe or purging device is rinsed with reagent water between samples. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of reagent water to check for cross contamination.

If sample or matrix interferences are encountered, a secondary or alternate analytical column may be used to resolve the compounds of interest.

2.2.1.3 General Laboratory Practices

A method blank consisting of organic free water spiked with surrogates and internal standards should be analyzed immediately following each daily calibration, and also after the analysis of every high concentration sample.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) analyses should be conducted to determine the effects of sample matrix upon the compounds of interest.

2.2.1.4 Sample Preparation

Method 5020 is a static headspace technique for extracting volatile organic compounds in pastes, solids, and liquids. Because of the large variability and complicated matrices of waste samples detection limits for this method may vary widely among samples.

Method 5030 is a purge-and-trap method applicable to nearly all types of samples, regardless of water content, including aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, groundwater, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments.

For highly volatile matrices, direct injection preceded by dilution should be used to prevent gross contamination of the instrumentation. For pastes, dilution of the sample until it becomes free-flowing is used to ensure adequate interfacial area. The success of this method depends on the level of interferences in the sample; results may vary due to the large variability and complicated matrices of solid waste samples.

2.2.1.5 Data Overview Prior to Validation

Before commencing validation, the reviewer must preview the associated Chain-of-Custody (COC) reports to determine:

- If the appropriate number of samples are present in the data package and if each sample was correctly analyzed for the parameters and methods specified.
- The identity of all associated field quality control blanks and field duplicate pairs.

Because many samples may have required dilutions, re-extractions and/or re-analyses, the validator should preview the data package contents to determine which analyses represent the better quality data.

Unless specifically directed by client protocol, never annotate the laboratory data package. Before beginning evaluation, prepare working copies (i.e. photocopies) of all Form I reports (including those for samples, laboratory method blanks and MS/MSD analyses) and all laboratory quality control summary forms (including all initial and continuing calibration summary statistics).

2.2.1.6 Technical Evaluation Summary

All data evaluations must be conducted in accordance with applicable USEPA Regional protocols and/or specific client contract requirements. The applicable documents must be referenced during the data evaluation process as this S.O.P. is only intended as a general procedure for the data validation tasks.

General parameters such as Data Completeness, Overall System Performance, Chromatographic Quality, Detection Limits and Compound Identification are evaluated concurrently with the parameters discussed in the following subsections.

2.2.1.6.1 Holding Times

Holding times are evaluated by reviewing the COC reports, the individual sample Form I reports, and the associated laboratory raw data. Holding times are calculated from date of collection to date of analysis.

The technical maximum holding time allowance for aqueous samples preserved with hydrochloric acid (HCL) is 14 days.

No technical holding times for solid matrices have been promulgated; a 14-day maximum holding time allowance is currently being used.

For unpreserved aqueous samples, generally a 7-day maximum holding time allowance for aromatic compounds, along with a 14-day maximum holding time allowance for chlorinated hydrocarbons is used.

Positive results in affected samples are generally qualified as estimated (J); nondetects (UJ). These results are biased low. Some USEPA Regions apply the bias qualifiers, L and UL, instead. If the holding times are exceeded by a factor of 2 or more, the holding time exceedance is considered to be gross and positive results are generally qualified as estimated (J); nondetects are generally considered to be unreliable and are qualified (R). Results for which the holding time was grossly exceeded are biased low.

2.2.1.6.2 Calibration

Check that an initial calibration was performed for each instrument used for analysis and that all calibrations were performed at all appropriate concentration levels.

In general, either the correlation coefficient (R) or the Percent Relative Standard Deviation (%RSD) is evaluated in the data validation. If the correlation coefficient is chosen by the laboratory, the calibration curve should be checked for linearity. Generally, associated sample data are qualified as estimated (J, UJ) if the calibration curve correlation coefficient is <0.995. Professional judgment should be used to qualify sample data in cases when sample results fall outside the linear portion of the calibration curve. If the %RSD is used, determine which compounds have Percent Relative Standard Deviations (%RSDs) >40% and between 20%-40%. Circle these noncompliances on your working copies of these Forms. Spot-check (i.e., recalculate) a few of the %RSDs to verify the laboratory's computation.

Determine which samples are affected by reviewing the continuing calibration forms. Determine which continuing calibrations are associated with which initial calibrations. Write the affected samples on your working copies of the appropriate continuing calibration forms. Spot-check (i.e., recalculate) a few of the %Ds to verify the laboratory's computation.

Review the continuing calibration form and the associated laboratory raw data. Determine which compounds have Percent Differences (%Ds) >30% and between 15%-30%; circle the noncompliances on your working copies of these forms.

Generally, positive results for compounds for which %RSD or %D exceeds 40% or 30%, respectively, are qualified as estimated (J); nondetects (UJ). Check the specific applicable data validation protocol for further guidance as there are some protocol which reject nondetects if the %RSD or %D is excessive. Bias for these results cannot be determined.

Generally, positive results for compounds for which %RSD is between 20%-40% or %D is between 15%-30% are qualified as estimated (J). Qualification of nondetects is protocol-specific. Follow the rules provided in the appropriate validation protocol.

2.2.1.6.3 Blank Contamination

When using the information given below and in the appropriate USEPA Regional Functional Guidelines, keep in mind that the validation action levels derived are sample-specific and must be adjusted for dilution, sample aliquot used for analysis, and sample moisture content (when applicable).

The rules for qualifying data based on the occurrence of blank contamination vary based on regional protocols; the guidelines provided in the appropriate protocol should be followed.

Generally the blank contamination review process is completed by first considering the maximum amount of a particular contaminant occurring in the laboratory method blanks. (Do not consider lab blanks run after high concentration samples for purposes of determining carryover as laboratory method blanks!). Then repeat the process for contaminants occurring in the associated field quality control blanks. Action levels for qualification (10X or 5X depending upon whether or not the contaminant is a common contaminant) are then set. The list of common contaminants may vary among protocols. Additionally, some hierarchy among the field quality control blanks apply and the manner in which the qualifiers are applied vary [i.e. use of (U) or (B); replacement by CRQL, etc.]. Refer to appropriate protocol for specific guidance.

2.2.1.6.4 Surrogates

Surrogates are evaluated by reviewing the laboratory data package Form II reports and the laboratory raw data. The quality control ranges are given on the laboratory data package Form IIs; circle any noncompliances on your working copies of these Forms.

All results for all compounds in an affected sample are qualified if any one of the surrogate spike compounds fails to meet the quality control criteria provided. Generally, for samples having a surrogate recovery < 10%, positive results are qualified as estimated (J), nondetects are rejected (R). These results are biased low. For samples having a surrogate recovery which is low but > 10%, positive results are generally qualified as estimated (J); nondetects (UJ). The bias qualifiers (L, UL) may be used instead, depending upon the specific USEPA Regional guidance. For samples having a surrogate recovery which is high, positive results are generally qualified as estimated (J, K) based on regional guidance; these results are biased high. Nondetects are not qualified based on high surrogate recoveries.

2.2.1.6.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

Generally, no data are qualified based upon MS/MSD results alone. If qualification does occur, generally only the result for that particular noncompliant compound is qualified in the original unspiked sample analysis. Refer to the applicable data validation protocol for specific procedures for evaluating MS/MSD analyses.

2.2.1.6.6 Other Considerations

Laboratory precision can be evaluated by comparing the unspiked sample results with MS/MSD analyses results for unspiked compounds. Consider nondetects and results reported at concentrations less than

the Contract Required Quantitation Limit (CRQL) to be in agreement. Use professional judgment in determining whether to qualify sample results based on the comparison.

Likewise, compare the positive compound results for field duplicate samples. Generally, the Relative Percent Difference (RPD) between field duplicate results for the aqueous matrix should be <35%; for soil matrix results, <50%. Qualification of the sample data is limited to the specific field duplicate pair. Positive results for compounds showing imprecision are qualified as estimated (J); nondetects (UJ). Bias for these results cannot be determined.

In some USEPA Regions, a "Percent Solids" rule applies. For example, if a sediment sample contains <50% solids in USEPA Region II, all associated data are considered to be estimated and are qualified accordingly. Follow the appropriate protocol guidance when applicable.

2.2.1.6.7 Quantitation

Verify and record the quantitation of at least one compound per analytical fraction. If no positive results are reported, use the MS/MSD data to confirm proper computation by the laboratory. Validator and laboratory quantitations must agree within 10 percent.

2.2.1.7 Deliverables Guidance

In addition to any specific USEPA Regional requirements (e.g. data validation memorandum, data summary spreadsheets, Regional worksheets), all laboratory data package quality control summary forms, sample Form I reports, method blank Form Is, and the Chain-of-Custody report must be given to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct (in accordance with the appropriate USEPA Regional or client requirements) and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

2.2.2 Semivolatiles (SW8040A, 8060, 8090, 8100)

2.2.2.1 Applicability

Method 8040A is used to determine the concentration of the following phenolic compounds in groundwater, liquid, and solid matrices:

Phenol
2-Chlorophenol
2,4-Dichlorophenol
2,6-Dichlorophenol
Trichlorophenols
Tetrachlorophenols
Pentachlorophenol
Cresols (methyl phenols)
4-Chloro-3-methylphenol
2,4-Dimethylphenol
2-Nitrophenol
4-Nitrophenol
2,4-Dinitrophenol
2-sec-Butyl-4,6-dinitrophenol (DNBP)

2-Cyclohexyl-4,6-dinitrophenol
2-Methyl-4,6-dinitrophenol

Method 8060 is used to determine the concentration of the following phthalate esters in groundwater, liquid, and solid sample matrices:

Benzyl butyl phthalate
Bis(2-ethylhexyl)phthalate
Di-n-butyl phthalate
Di-n-octyl phthalate
Diethyl phthalate
Dimethyl phthalate

Method 8090 is used to determine the concentration of the following nitroaromatic and cyclic ketone compounds in groundwater, liquid, and solid sample matrices:

Nitrobenzene
Dinitrobenzene
2,4-Dinitrotoluene
2,6-Dinitrotoluene
Isophorone
Naphthoquinone

Method 8100 is used to determine the concentration of the following polynuclear aromatic hydrocarbons (PAHs) in liquid and solid sample matrices:

Acenaphthene
Acenaphthylene
Anthracene
Benzo(a)anthracene
Benzo(a)pyrene
Benzo(b)fluoranthene
Benzo(ghi)perylene
Benzo(j)fluoranthene
Benzo(k)fluoranthene
Chrysene
Dibenzo(a,h)anthracene
Dibenz(a,h)acridine
Dibenz(a,j)acridine
7H-Dibenzo(c,g)carbazole
Dibenzo(a,e)pyrene
Dibenzo(a,h)pyrene
Dibenzo(a,i)pyrene
Fluoranthene
Fluorene
Indeno(1,2,3-cd)pyrene
3-Methylcholanthrene
Naphthalene
Phenanthrene
Pyrene

All of the above methods are gas chromatographic (GC) only (i.e., no mass spectrometer detector is employed). These methods use either an electron capture detector (ECD) or a flame ionization detector (FID).

2.2.2.2 Interferences

Solvents, reagents, glassware, and other sample-processing hardware may yield discrete artifacts and/or elevated baselines causing misinterpretation of gas chromatograms. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by running method blanks. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be required.

Interferences co-extracted from samples will vary considerably from source to source depending upon the waste being sampled. While general cleanup techniques such as Method 3530 are provided as part of these methods, unique samples may require additional cleanup.

If sample or matrix interferences occur, a secondary column may be employed in addition to the primary column so as to resolve any questionable compound results.

2.2.2.3 General Laboratory Practices

An extraction blank should be prepared with each batch of samples extracted.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) analyses should be conducted to determine the effects of sample matrix upon the compounds of interest.

2.2.2.4 Sample Preparation

Prior to analysis, the samples must be extracted using the appropriate techniques. Aqueous samples are extracted at the appropriate pH with methylene chloride as a solvent using Method 3510 (separatory funnel extraction) or Method 3520 (continuous liquid-liquid extraction). Both neat and diluted organic liquids may be analyzed by direct injection. Solid samples are extracted at the appropriate pH with methylene chloride using either Soxhlet Extraction (Method 3540) or Sonication (Method 3550) procedures.

2.2.2.5 Data Overview Prior to Validation

Before commencing validation the reviewer must preview the associated Chain-of-Custody (COC) reports to determine:

- If the appropriate number of samples are present in the data package and if each sample was correctly analyzed for the parameters and methods specified.
- The identity of all associated field quality control blanks and field duplicate pairs.

Because many samples may have required dilutions, re-extractions and/or reanalyses, the data validator should preview the data package contents to determine which analyses represent the better quality data.

The data package should never be annotated unless specifically directed by client protocol. All Form I reports (including those for samples, laboratory method blanks, and MS/MSD analyses) and all

laboratory quality control summary forms (including all initial and continuing calibration summary statistics) should be photocopied for use as working copies.

2.2.2.6 Technical Evaluation Summary

All data evaluations must be conducted in accordance with the appropriate USEPA Regional protocols and/or specified client contract requirements. The applicable documents must be referenced during the data validation process as this S.O.P. is only intended as a general procedure for the data validation tasks.

General parameters such as Data Completeness, Overall System Performance, Chromatographic Quality, Detection Limits and Compound Identification are evaluated concurrently with the parameters discussed in the following subsections.

2.2.2.6.1 Holding Times

Holding times are evaluated by reviewing the COC reports, the individual sample Form I reports, and the associated laboratory raw data. Holding times for extraction are calculated from date of collection to date of extraction.

The technical holding times for aqueous and solid matrices are as follows:

- Extraction:
  - Water samples: 7 days
  - Solid samples: 14 days
- Analysis: 40 days from date of extraction

Generally, positive results affected by noncompliances are qualified as estimated (J); nondetects (UJ). These results are considered to be biased low. Alternately, the bias qualifiers L and UL may be used. Nondetects may be rejected (R) when the sample was extracted after 14 days (28 days for solid samples). If the holding time until analysis has been exceeded (and potentially, some of the extract may have evaporated), the affected sample results may be considered to be biased high. Refer to the appropriate data validation protocol for specific guidance.

2.2.2.6.2 Calibration

Check that an initial calibration was performed for each instrument used for analysis and that all calibrations were performed at all appropriate concentration levels.

In general, either the correlation coefficient (R) or the Percent Relative Standard Deviation (%RSD) is evaluated in the data validation. If the correlation coefficient is chosen by the laboratory, the calibration curve should be checked for linearity. Generally, associated sample data are qualified as estimated (J, UJ) if the calibration curve correlation coefficient is <0.995. Professional judgment should be used to qualify sample data in cases when sample results fall outside the linear portion of the calibration curve. If the %RSD is used, determine which compounds have Percent Relative Standard Deviations (%RSDs) >40% and between 20%-40%. Circle these noncompliances on your working copies of these Forms. Spot-check (i.e., recalculate) a few of the %RSDs to verify the laboratory's computation.

Determine which samples are affected by reviewing the continuing calibration forms. Determine which continuing calibrations are associated with which initial calibrations. Write the affected samples on your

working copies of the appropriate continuing calibration forms. Spot-check (i.e., recalculate) a few of the %Ds to verify the laboratory's computation.

Review the continuing calibration form and the associated laboratory raw data. Determine which compounds have Percent Differences (%Ds) >30%, and between 15%-30%; circle the noncompliances on your working copies of these forms.

Generally, positive results for compounds for which %RSD or %D exceeds 40% or 30%, respectively, are qualified as estimated (J); nondetects (UJ). Check the specific applicable data validation protocol for further guidance as there are some protocol which reject nondetects if the %RSD or %D is excessive. Bias for these results cannot be determined.

Generally, positive results for compounds for which %RSD is between 20%-40% or whose %D is between 15%-30% are qualified as estimated (J). Qualification of nondetects is protocol-specific. Follow the rules provided in the appropriate validation protocol.

2.2.2.6.3 Blank Contamination

When using the information given below and in the appropriate USEPA Regional Functional Guidelines, keep in mind that the validation action levels derived are sample-specific and must be adjusted for dilution, sample aliquot used for analysis, and sample moisture content (when applicable).

The rules for qualifying data based on the occurrence of blank contamination vary based on regional protocols; the guidelines provided in the appropriate protocol should be followed.

Generally the blank contamination review process is completed by first considering the maximum amount of a particular contaminant occurring in the laboratory method blanks. (Do not consider lab blanks run after high concentration samples for purposes of determining carryover as laboratory method blanks!) Then repeat the process for contaminants occurring in the associated field quality control blanks. Action levels for qualification (10X or 5X depending upon whether or not the contaminant is a common contaminant) are then set. The list of common contaminants may vary among protocols. Additionally, some hierarchy among the field quality control blanks apply and the manner in which the qualifiers are applied vary [i.e. use of (U) or (B); replacement by CRQL, etc.]. Refer to appropriate protocol for specific guidance.

2.2.2.6.4 Surrogates

Surrogates are evaluated by reviewing the laboratory data package Form II reports and the laboratory raw data. The quality control ranges are given on the laboratory data package Form IIs; circle any noncompliances on your working copies of these Forms.

All results for all compounds in an affected sample are qualified if any one of the surrogate spike compounds fails to meet the quality control criteria provided. Generally, for samples having a surrogate recovery <10%, positive results are qualified as estimated (J), nondetects are rejected (R). These results are biased low. For samples having a surrogate recovery which is low but >10%, positive results are generally qualified as estimated (J); nondetects (UJ). The bias qualifiers (L, UL) may be used instead, depending upon the specific USEPA Regional guidance. For samples having a surrogate recovery which is high, positive results are generally qualified as estimated (J, K) based on regional guidance; these results are biased high. Nondetects are not qualified based on high surrogate recovery.

2.2.2.6.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

Generally, no data are qualified based upon MS/MSD results alone. If qualification does occur, generally only the result for that particular noncompliant compound is qualified in the original unspiked sample. Refer to the applicable data validation protocol for specific procedures for evaluating MS/MSD analyses.

2.2.2.6.6 Other Considerations

Laboratory precision can be evaluated by comparing the unspiked sample results with MS/MSD analyses results for unspiked compounds. Consider nondetects and results reported at concentrations less than the Contract Required Quantitation Limit (CRQL) to be in agreement. Use professional judgment in determining whether to qualify sample results based on the comparison.

Likewise, compare the positive compound results for field duplicate samples. Generally, the Relative Percent Difference (RPD) between field duplicate results for the aqueous matrix should be <35%; for soil matrix results, <50%. Qualification of the sample data is limited to the specific field duplicate pair. Positive results for compounds showing imprecision are qualified as estimated (J); nondetects (UJ). Bias for these results cannot be determined.

In some USEPA Regions, a "Percent Solids" rule applies. For example, if a sediment sample contains <50% solids in USEPA Region II, all associated data are considered to be estimated, and are qualified accordingly. Follow the appropriate protocol guidance when applicable.

2.2.2.6.7 Quantitation

Verify and record the quantitation of at least one compound per analytical fraction. If no positive results are reported, use the MS/MSD data to confirm proper computation by the laboratory. Validator and laboratory quantitations must agree within 10 percent.

2.2.2.7 Deliverables Guidance

In addition to any specific USEPA Regional requirements (e.g. data validation memorandum, data summary spreadsheets, Regional worksheets), all laboratory data package quality control summary forms, sample Form I reports, method blank Form Is, and the Chain-of-Custody report, must be given to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct (in accordance with the appropriate USEPA Regional or client requirements), and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

2.2.3 Organochlorine Pesticides and Polychlorinated Biphenyls (PCBs), Organophosphorous Pesticides, Chlorinated Herbicides (SW 8080B, 8140, 8150B)

2.2.3.1 Applicability

Method 8080B is used to determine the concentration of the following organochlorine pesticides and polychlorinated biphenyls (PCBs) in groundwater, liquid, and solid sample matrices:

Aldrin
alpha-BHC
beta-BHC

delta-BHC
gamma-BHC (Lindane)
Chlordane
4,4'-DDD
4,4'-DDE
4,4'-DDT
Dieldrin
Endosulfan I
Endosulfan II
Endosulfan sulfate
Endrin
Endrin aldehyde
Heptachlor
Heptachlor epoxide
Methoxychlor
Toxaphene
Aroclor-1016
Aroclor-1221
Aroclor-1232
Aroclor-1242
Aroclor-1248
Aroclor-1254
Aroclor-1260

Similarly, Method 8140 is used to determine the following pesticides in groundwater and waste samples:

Azinphos methyl
Bolstar (Sulprofos)
Chlorpyrifos
Coumaphos
Demeton-O
Demeton-S
Diazinon
Dichlorvos
Disulfoton
Ethoprop
Fensulfothion
Fenthion
Merphos
Mevinphos
Naled
Parathion methyl
Phorate
Ronnel
Stirophos (Tetrachlorvinphos)
Tokuthion (Prothiofos)
Trichloronate

Note that when Method 8140 is used to analyze unfamiliar samples, compound identifications should be supported by at least one additional qualitative technique if mass spectroscopy is not employed.

Method 8150B is used to determine the following chlorinated acid herbicides in groundwater and waste samples:

2,4-D
2,4-DB
2,4,5-T
2,4,5-TP (Silvex)
Dalapon
Dicamba
Dichloroprop
Dinoseb
MCPA
MCPP

Since these compounds are produced and used in various forms (i.e., acid, salt, ester, etc.), Method 8150 includes a hydrolysis step to convert the herbicide to the acid form prior to analysis. When Method 8150 is used to analyze unfamiliar samples, compound identifications should be supported by at least one additional qualitative technique. This method describes analytical conditions for a second gas chromatographic column that can be used to confirm measurements made with the primary column; alternately, the compounds of interest can be confirmed by detection via a mass spectrometer.

All of the above Methods are Gas Chromatographic (GC) in which sample extracts are analyzed by direct injection. Method 8080 analyzes for organochlorine pesticide compounds and PCBs via GC/ECD (Electron Capture Detector; an equivalent Halogen-Specific Detector may also be used). Method 8140 analyzes for organophosphorous pesticide compounds via GC/FID (Flame Ionization Detector), and Method 8150 analyzes for chlorinated herbicide compounds via GC/ECD (alternately, a Microcoulometric Detector or Hall Electrolytic Conductivity Detector may be used).

2.2.3.2 Interferences

The sensitivity of these methods usually depends on the level of interferences rather than on instrumental limitations. Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated baselines causing misinterpretation of gas chromatograms. The use of high purity reagents and solvents helps to minimize these interference problems. Extraction blanks are analyzed as method blanks in order to monitor the occurrences of interferences.

Interferences co-extracted from the sample will vary considerably, and will dictate the nature and extent of clean-up procedures used. Phthalate esters are a common interference to organochlorine pesticide analyses; phenols and organic acids may act as interferents when analyzing for chlorinated herbicides.

2.2.3.3 General Laboratory Practices

Matrix Spike/Matrix Spike Duplicate (MS/MSD) analyses should be conducted to determine the effects of sample matrix upon the compounds of interest.

Standard quality assurance practices such as the analyses of field replicate and laboratory duplicates should also be employed.

Note that herbicides, being strong organic acids, react readily with alkaline substances and may be lost during analysis. Therefore, when performing Method 8150, glassware and glass wool must be acid-rinsed and sodium sulfate must be acidified with sulfuric acid prior to use to avoid this possibility.

2.2.3.4 Sample Preparation

Prior to the use of Method 8080 and 8140, aqueous samples are extracted at a neutral pH with methylene chloride as a solvent using a separatory funnel (Method 3510) or a continuous liquid- liquid extractor (Method 3520). Solid samples are extracted with hexane:acetone (1:1) using either the Soxhlet extraction (Method 3540) or sonication (Method 3550) procedures.

Method 8150 provides its own specific preparation procedures for aqueous and solid samples which include extraction with acetone and diethyl ether followed by esterification using diazomethane as a derivatizing agent.

2.2.3.5 Data Overview Prior to Validation

Before commencing validation, the reviewer must preview the associated Chain-of-Custody (COC) reports to determine:

- If the appropriate number of samples are present in the data package and if each sample was correctly analyzed for the parameters and methods specified.
- The identity of all associated field quality control blanks and field duplicate pairs.

Because many samples may have required dilutions, re-extractions and/or re-analyses, the validator should preview the data package contents to determine which analyses represent the better quality data.

Unless specifically directed by client protocol, never annotate the laboratory data package. Before beginning evaluation, prepare working copies (i.e., photocopies) of all Form I reports (including those for samples, laboratory method blanks and MS/MSD analyses) and all laboratory quality control summary forms.

2.2.3.6 Technical Evaluation Summary

All data evaluations must be conducted in accordance with applicable USEPA Regional protocols and/or specific client contract requirements. The applicable documents must be referenced during the data evaluation process as this S.O.P. is only intended as a general procedure for the data validation tasks.

General parameters such as Data Completeness, Overall System Performance, Chromatographic Quality, Detection Limits, and Compound Identification are evaluated concurrently with the parameters discussed in the following subsections.

2.2.3.6.1 Holding Times

Holding times are evaluated by reviewing the COC reports, the individual sample Form I reports, and the associated laboratory raw data. Holding times for extraction are calculated from date of collection to date of extraction.

The technical holding times for aqueous and solid matrices are as follows:

- Extraction:
  - Water samples: 7 days
  - Solid samples: 14 days
- Analysis: 40 days from date of extraction

When the holding time criteria are not met, positive results in affected samples are generally qualified as estimated (J); nondetects (UJ). These results are biased low. Some USEPA Regions apply the bias qualifiers, L and UL, instead. If the holding times are exceeded by a factor of 2 or more, the holding time exceedance is considered to be gross and positive results are generally qualified as estimated (J); nondetects are generally considered to be unreliable and are rejected (R). These results are biased very low.

2.2.3.6.2 Calibration

Data pertaining to the initial calibration (i.e., evaluation check for linearity) is found on the data package Form VIs or equivalent. Check that an initial calibration was performed for each instrument used and at all appropriate concentration levels.

In general, either the correlation coefficient (R) or the Percent Relative Standard Deviation (%RSD) is evaluated in the data validation. If the correlation coefficient is chosen by the laboratory, the calibration curve should be checked for linearity. Generally, associated sample data are qualified as estimated (J, UJ) if the calibration curve correlation coefficient is <0.995. Professional judgment should be used to qualify sample data in cases when sample results fall outside the linear portion of the calibration curve. If the %RSD is used, determine which compounds have Percent Relative Standard Deviations (%RSDs) >40% and between 20%-40%. Circle these noncompliances on your working copies of these Forms. Spot-check (i.e., recalculate) a few of the %RSDs to verify the laboratory's computation.

Determine which samples are affected by reviewing the continuing calibration forms. Determine which continuing calibrations are associated with which initial calibrations. Write the affected samples on your working copies of the appropriate continuing calibration forms. Spot-check (i.e., recalculate) a few of the %Ds to verify the laboratory's computation.

Review the continuing calibration form and the associated laboratory raw data. Determine which compounds have Percent Differences (%Ds) >30% and between 15%-30%; circle the noncompliances on your working copies of these forms.

Generally, positive results for compounds for which %RSD or %D exceeds 40% or 30%, respectively, are qualified as estimated (J); nondetects (UJ). Check the specific applicable data validation protocol for further guidance as there are some protocol which reject nondetects if the %RSD or %D is excessive. Bias for these results cannot be determined.

Generally, positive results for compounds for which %RSD is between 20%-40% or %D is between 15%-30% are qualified as estimated (J). Qualification of nondetects is protocol-specific. Follow the rules provided in the appropriate validation protocol.

Method 8080A requires analysis of a DDT/Endrin breakdown check standard. The DDT/Endrin Breakdown should not exceed 20%. Generally, if % breakdown for DDT exceeds 20%, estimate (J) all positive results for DDT, DDE and DDD following the in-last control standard until the next in-control standard (see analytical sequence). If there are no positive results for DDT but there are positive results for DDD or DDE then reject (R) nondetects for DDT in associated samples. Generally, if Endrin % Breakdown exceeds 20%, estimate (J) positive results for Endrin, Endrin Aldehyde, and Endrin Ketone in all samples following the last in-control standard until the next acceptable standard. If there are positive results for Endrin Aldehyde or Endrin Ketone but none for Endrin, reject (R) nondetect Endrin results.

2.2.3.6.3 Blank Contamination

When using the information provided below and in the appropriate USEPA Regional Functional Guidelines, keep in mind that the validation action levels derived are sample-specific, and must be adjusted for dilution, sample aliquot used for analysis, and sample moisture content (when applicable).

The rules for qualifying data based on the occurrence of blank contamination vary based on regional protocols; guidelines provided in the appropriate data validation protocol should be followed.

An action level of 5X the maximum amount of contaminant found is used to evaluate the sample data. The manner in which the qualifiers are applied vary [i.e. use of (U) or (B); replacement by CRQL, etc.]. Refer to appropriate validation protocol for specific guidance.

2.2.3.6.4 Surrogates

Surrogates are evaluated by reviewing the laboratory data package Form II reports and the associated laboratory raw data. The advisory limits are given on the laboratory data package Form IIs; circle any recoveries outside these limits on your working copies of these Forms.

No qualifications are made for surrogates which show zero recoveries because they were "diluted out." Generally, positive results affected by low surrogate recovery are qualified as estimated (J) or the (L) bias qualifier is used when applicable; nondetects are qualified (UJ) or (UL), accordingly. If a positive sample result is affected by high surrogate recovery, the result is qualified as estimated (J) or the (K) bias qualifier is used when applicable; nondetects are not qualified based on high surrogate recovery. Because the surrogate recovery limits for these fractions are advisory, generally no results are rejected.

The pesticide/PCB surrogates decachlorobiphenyl (DCB) and tetrachloro-m-xylene (TCX) retention times found on data package Form VIII or equivalent must be ±0.10 for DCB and ±0.05 for TCX. If DCB and TCX retention time criteria are not met, the raw data must be checked for misidentified GC peak. The validator's professional judgment for qualifications should be used.

2.2.3.6.5 Matrix Spike/Matrix Spike Duplicates

Generally, no data are qualified based upon MS/MSD results alone. If qualification does occur, generally only the result for that particular noncompliant compound is qualified in the original unspiked sample analysis. Refer to the appropriate data validation guidelines for specific procedures for evaluating MS/MSD analyses.

2.2.3.6.6 Other Considerations

Laboratory precision can be evaluated by comparing the unspiked sample results with MS/MSD analyses results for unspiked compounds. Consider nondetects and results reported at concentrations less than the Contract Required Quantitation Limit (CRQL) to be in agreement. Use professional judgment in determining whether to qualify sample results based on the comparison.

Likewise, compare the positive compound results for field duplicate samples. Generally, the Relative Percent Difference (RPD) between field duplicate results for the aqueous matrix should be <35%; for soil matrix results, <50%. Qualification of the sample data is limited to the specific field duplicate pair. Positive results for compounds showing imprecision are qualified as estimated (J); nondetects (UJ). Bias for these results cannot be determined.

In some USEPA Regions, a "Percent Solids" rule applies. For example, if a sediment sample contains <50% solids in USEPA Region II, all associated data are considered to be estimated and are qualified accordingly. Follow the appropriate protocol guidance when applicable.

2.2.3.6.7 Quantitation

Verify and record the quantitation of at least one compound per analytical fraction. If no positive results are reported, use the MS/MSD data to confirm proper computation by the laboratory. Validator and laboratory quantitations must agree within 10%.

2.2.3.7 Deliverables Guidance

In addition to any specific USEPA Regional requirements (e.g. data validation memorandum, data summary spreadsheets, USEPA Regional worksheets), all laboratory data package quality control summary forms, sample Form I reports, method blank Form Is, and the Chain-of-Custody report, must be given to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct (in accordance with the appropriate USEPA Regional or client requirements), and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

3.0 CLP INORGANICS FOR SOLID AND AQUEOUS MATRICES

3.1 Inorganics (CLP Statement of Work (SOW) ILM03.0)

Inductively Coupled Plasma Emission Spectroscopy (ICP) - Analytes commonly analyzed using ICP include: aluminum, barium, beryllium, cadmium, calcium, chromium, cobalt, copper, iron, magnesium, manganese, nickel, potassium, silver, sodium, vanadium, and zinc.

Graphite Furnace Atomic Absorption Spectroscopy (GFAA) - Analytes commonly analyzed using GFAA include: antimony, arsenic, lead, selenium, and thallium

Cold Vapor Methodology - Mercury is commonly analyzed using cold vapor methodology.

Automated Colorimetric Technique - Cyanide is commonly analyzed using automated colorimetric methodology.

3.1.1 Applicability

This method is applicable to a large number of matrices including EP extracts, TCLP extracts, industrial wastes, soils, groundwater, aqueous samples, sludges, sediments, and other solid wastes. All matrices require digestion prior to analysis.

3.1.2 Data Overview Prior to Validation Process

3.1.2.1 Data Completeness

The data reviewer must initially verify that all CLP Forms are present and complete (i.e., Forms 1 through 14 must be provided). Areas of special attention when accounting for required CLP Forms will include:

- Verify at least one Initial and Continuing Calibration Verification (ICV/CCV) Percent Recovery (%R) calculation as noted on the Form 2A.

- When reviewing Form 2B, verify that all atomic absorption (GFAA) analytes are present in the CRDL standard at concentrations at the CRDL. Verify that all ICP analytes (with the exceptions of Al, Ba, Ca, Fe, Mg, Na and K) are present in the CRDL standard at concentrations of 2X CRDL.

- Verify that a matrix-specific laboratory generated preparation blank has been analyzed for each respective matrix as noted on the Form 3 (note that filtered and unfiltered aqueous matrices are to be treated as distinctly different matrices).

- Verify that all ICP analytes are present in both ICSA and ICSAB solutions. (Note that 3/90 SOW ILM03.0 does not require that antimony, sodium, and potassium be present in these solutions). Also verify from the raw data that the laboratory reported all analytes present in solution A to the nearest whole number. It is not uncommon for laboratories to incorrectly report "zeros" or simply leave blank the appropriate solution A columns. Furthermore, %Rs for solution AB are to be reported to one decimal place on the Form 4.

- Check that one matrix spike was analyzed for each particular matrix per analytical batch. Laboratories typically will not include an aqueous matrix for waters if the only aqueous samples contained in the SDG are field quality control blanks (i.e., equipment rinsate blanks

and/or field blanks). This is generally accepted without data validation letter text comment. Additionally, the data reviewer may want to verify spiking levels as noted on pg. E-20 of ILM03.0 3/90 Inorganic SOW.

- Verify that laboratory duplicate analyses were performed for each matrix. **NOTE**: Field quality control blanks are never to be designated for quality control analyses.

- Check that one Laboratory Control Sample (LCS) was analyzed for each batch of samples per matrix within an SDG. **NOTE**: An aqueous LCS is not required for mercury and cyanide analysis.

- The Method of Standard Additions (MSA) Form 8 may or may not be present as dictated by Post Digestion Spike (PDS) %Rs. See Section 3.1.3.11 for further details.

- Verify that at least one ICP serial dilution analysis was performed for each matrix within an SDG. **NOTE**: Typically one serial dilution will serve to monitor a given set of samples within an SDG. However, special contractual requirements may necessitate one serial dilution analysis per sample. Ascertain atypical serial dilution frequency requirements through the project manager.

- Simply check that the Form 11 ICP Interelement Correction Factors (Annually) is present.

- Verify that all ICP analytical results fall within the ICP Quarterly Linear Ranges provided on the Form 12. Verify that no GFAA analytical results exceed the highest standard used in the associated GFAA calibration.

- Verify that the Form 13 Preparation Log accounts for aqueous/soil ICP, AA, mercury, and cyanide digestions/distillations as applicable.

- Examine the Form 14s to verify that one and only one "X" flag has been used to signify each reported field sample result or quality control sample result. Laboratories are often careless when entering the "X" flag. An incorrectly entered "X" flag can lead to reporting errors for the sample and its associated QC. The validator must verify reported results in instances of discrepancies, amend appropriate forms, and mention in letter text.

Actions - Notify the appropriate laboratory contact of required resubmittals when discrepancies are noted on the forms discussed above.

3.1.3 Technical Evaluation Summary

All data evaluations must be conducted in accordance with current and applicable USEPA Regional protocols and/or specific client contractual requirements and obligations. The applicable documents must be referenced to during the data evaluation process as this Standard Operating Procedure (S.O.P) is intended as proprietary in-house guidance for general inorganic validation practices only.

General parameters such as Data Completeness, Overall System Performance, and Detection Limits must be evaluated concurrently with the parameters discussed below.

3.1.3.1 Holding Times

Holding times are calculated from date of sample collection to date of sample analysis. The date of sample collection must be obtained from the Chain-of-Custody (COC) form. The date of sample analysis is best retrieved from the raw data but may also be obtained from the Form 14.

Sample preservation and holding time requirements are as follows:

- Metals - 6 months; pH <2
- Mercury - 28 days; pH <2
- Cyanide - 14 days; pH >12

Preservation requirements as noted above are applicable to aqueous samples only; solid samples do not receive preservative, but require maintenance at 4°C (±2°C) during shipment and storage.

Actions - Holding time exceedances result in potentially low-biased results; thus, positive results and nondetects shall be qualified as estimated, (J) and (UJ), respectively. **NOTE**: Gross holding time noncompliances are defined as holding times which are exceeded by a factor or 2X. In these extreme cases, it is practice to reject (R) nondetects while positive results are qualified based upon professional judgment regarding the reliability of the associated data.

3.1.3.2 Initial Calibration Requirements

Calibration must be initiated daily and prior to sample analysis. The following calibration standard requirements must be verified:

- **ICP analyses** - must employ a blank and at least one standard.
- **GFAA analyses** - must employ a blank and at least three standards. One of the standards must be at the CRDL. Additionally, the calibration correlation coefficient (r) must be checked for linearity for each GFAA analysis performed (i.e., r = 0.995 or greater).
- **Mercury analyses** - must employ a blank and at least four standards (r = 0.995 or greater).
- **Cyanide analyses** - must employ a blank and at least three standards (r = 0.995 or greater). **NOTE:** The midpoint standard for cyanide analyses must be distilled; verify this via distillation logs.

3.1.3.3 Initial and Continuing Calibration Verification (ICV/CCV)

Review Initial and Continuing Calibration Verification Form 2As and associated raw data. The ICV/CCV %R quality control limits are 90-110% for metals, 80-120% for mercury, and 85-115% for cyanide.

Actions - If ICV/CCV %Rs are low, qualify as estimated (J) positive results and (UJ) nondetects. If ICV/CCV %Rs are high, qualify as estimated (J) positive results; nondetects are not affected. Gross exceedance, as defined by applicable data validation protocol, may require rejection (R) of results. **NOTE:** Qualify results of only those samples associated with the noncompliant ICB or CCV (generally, those samples immediately preceding or following the noncompliant standard until the nearest in-control standard).

3.1.3.4 CRDL Standard Analysis

Review CRDL Standard Form 2Bs and associated new data. The CRDL Standard analysis %R quality control limits are 80-120% for all metals.

Actions - If CRDL %Rs are low, qualify as estimated (J) positive results <3X CRDL and (UJ) nondetects. Generally, if CRDL %Rs are high, qualify as estimated (J) positive results <3X CRDL; nondetects remain unaffected. Note that when using EPA Region I validation guidelines, nondetects will receive qualification based upon high CRDL Standard analysis recovery. **NOTE:** The data reviewer need not specify affected samples; common practice is to apply data qualifications "across-the-board" based upon LOE time constraints.

3.1.3.5 Laboratory Method and Field Quality Control Blanks

Verify that a preparation blank was analyzed for each matrix and for each batch of 20 samples or each sample batch digested, whichever is more frequent. Continuing Calibration Blanks (CCBs) must be run at a frequency of 10% or every 2 hours whichever is more frequent.

The data reviewer will select the maximum contaminant level for each analyte in a particular matrix from which shall be calculated an "action level." The action level shall be established as 5X the maximum contaminant level but must be adjusted for dilution factor, moisture content, and sample weight prior to application.

ICB/CCB contamination shall be applied to all samples within an SDG. Preparation blank contamination shall be applied to samples of the same matrix only. Common practice shall be to qualify as nondetected (U) any contaminant present in sample which is considered a laboratory artifact (i.e., < the established action level). Professional judgment must be employed when discerning the validity of a concentration present in a field quality control blank. In many instances, contamination present in these blanks can be attributable to "dirty" laboratory practice and not actual field contaminant conditions.

Negative concentrations detected in the laboratory method blanks are indicative of instrumental problems and base-line drifting. Generally, any negative concentration > IDL shall warrant estimation [(J) positives and (UJ) nondetects] of the associated sample data regardless of matrix. Action levels shall not be established for negative concentration levels.

Actions - Qualify as nondetected (U) any positive result within the action level. Qualify as estimated (J) positive results and (UJ) nondetects for analytes for which negative concentrations were noted in the laboratory method blanks (i.e., ICBs, CCBs, and/or preparation blanks).

3.1.3.6 ICP Interference Check Sample (ICS) Results

Review ICP Interference Check Sample Form 4 and associated raw data. Verify that all recoveries for the ICP ICS solution fall within the 80-120% quality control window established for the ICS AB solution.

Actions - For ICS %Rs <80%, qualify as estimated (J) positive results and (UJ) nondetects in affected samples. For ICS %Rs >120%, qualify as estimated (J) positive results in affected samples; nondetects are unaffected by high ICS solution AB recovery. **NOTE:** Affected samples include all samples analyzed between the initial and final solutions (or within the eight hour working shift, whichever occurs more frequently) which contain Al, Ca, Fe, or Mg at levels >50% of the respective concentration of Al, Ca, Fe, or Mg in the ICS True Solution A.

Next, review concentrations of the four common interfering analytes (aluminum, calcium, iron, and magnesium) in the environmental samples. Any aforementioned interferant present in the environmental samples at concentrations which exceed 50% of those present in the ICS solution for that same analyte will require calculation of estimated elemental interference stemming from high interfering analyte concentration. If the previous condition is met; review the ICP/ICS Form 4 and note any analytes present in the ICS solution A at levels which exceed the IDL and which are not present in the ICS True solution A. Positive results in the ICS solution A indicate potentially elevated results for this analyte in the affected sample while negative results in the ICS solution A indicate potentially suppressed results for this analyte in the affected sample.

Next, an estimated elemental interference must be calculated for each analyte > IDL present in the ICS solution A which is not present in the ICS True solution A. The following equation shall be employed:

$$\textit{Estimated elemental intf.} = \frac{[\textit{Conc. affected analyte in ICS Soln A}] \times [\textit{Interferent}]\ [\textit{Conc. in Sample}]}{\textit{Interferent Conc. in ICS Soln A}}$$

It is advisable, although not necessary, to routinely choose the lowest concentration for the interferant level in the ICS so as to calculate the highest estimated interference possible. This method lends itself to a more conservative overall data quality review.

Estimated interferences for each affected analyte > IDL in the ICSA solution must now be compared to the reported environmental sample result for that particular analyte.

Actions - For estimated interferences <10% of the reported sample concentration for a particular affected analyte, take no action; interference is considered negligible. For estimated interferences >10% of the reported sample concentration for a particular affected analyte, qualify (J) positive result and/or (UJ) nondetect for affected analyte in affected sample. (**NOTE**: Calculation of an estimated positive (potentially elevated) interference will have no effect on a reported nondetect; thus, no action is necessary).

3.1.3.7 Matrix Spike Sample Analysis (Pre-digestion)

Review Spike Sample Recovery Form 5A and associated raw data. Verify that at least one matrix spike was performed for each matrix for a given set of samples within an SDG. **NOTE:** Filtered and unfiltered samples are to be treated as distinctly different sample matrices and qualified accordingly. Refer to ILM03.0, 3/90 Inorganic SOW, Table 3, "SPIKING LEVELS FOR SPIKING SAMPLE ANALYSIS," page 20, Section E, for proper analyte spiking concentrations and requirements. Any deviations from the SOW shall be noted and require laboratory contact for correction.

Aqueous and soil Matrix Spike (MS) recoveries must be within the 75-125% quality control window in instances where the initial sample result is <4X amount spiked. If the initial sample result is >4X the amount spiked and the MS %R is noncompliant; no actions shall be taken.

Actions - For MS %Rs <30%, qualify as estimated (J) positive results and reject (R) nondetects in affected samples. For MS %Rs <75% but >30%, qualify as estimated (J) positive results and (UJ) nondetects in affected samples. For MS %Rs >125%, qualify as estimated (J) positive results in affected samples; nondetects are not compromised by high MS recovery; thus, no actions are warranted.

3.1.3.8 Laboratory Duplicate Precision

Review Duplicates Form 6 and associated raw data. Verify that one duplicate sample analysis was performed for each group of samples of a similar matrix within an SDG. Control criteria used to evaluate aqueous laboratory duplicates are as follows:

- a control limit of ±20% for relative percent difference when sample and duplicate results are >5X CRDL
- a control limit of ±1X CRDL for the difference between the sample values when sample and/or duplicate results are <5X CRDL

Control criteria used to evaluate solid laboratory duplicates are as follows:

- a control limit of ±35% for relative percent difference when sample and duplicate results are >5X CRDL
- a control limit of ±2X CRDL for the difference between the sample values when sample and/or duplicate results are <5X CRDL

NOTE: Review the CLP Form 6 carefully and verify that the laboratory has in fact reported a %RPD of 200% and not simply recorded the %RPD as noncalculable (in instances where the sample result is positive but the duplicate result is nondetect). Overlooking this minor point may result in incomplete sample data qualification in some instances.

Actions - For any situation involving laboratory duplicate imprecision, qualify as estimated (J) positive results and (UJ) nondetects in affected samples. **NOTE:** It is important to note in the letter text the cause of laboratory duplicate imprecision (i.e., noncompliant %RPD or noncompliant difference between sample and duplicate results).

3.1.3.9 Field Duplicate Precision

Field duplicates can be determined via Project Manager informational documents (i.e., sampling logs) or obtained from Chain-of-Custody (COC) forms. Field duplicates are generally identified as samples having identical sample collection times and dates. In instances were field duplicate samples are included with the sample data set, the following control criteria are generally used to evaluate aqueous field duplicates:

- a control limit of ±30% for relative percent difference when sample and duplicate results are >5X CRDL
- a control limit of ±2X CRDL for the difference between the sample values when sample and/or duplicate results are <5X CRDL

Similarly, the following control criteria are generally used to evaluate solid field duplicates:

- a control limit of ±50% for the relative percent difference when sample and duplicate results are >5X CRDL
- a control limit of ±4X CRDL for the difference between the sample values when sample and/or duplicate results are <5X CRDL

NOTE: The %RPD should reflect a difference of 200% and should not simply be recorded as noncalculable in instances where the sample result is positive but the field duplicate result is nondetect. Overlooking this minor point may result in incomplete sample data qualification in some instances.

Actions - For any situation involving field duplicate imprecision, qualify as estimated (J) positive results and (UJ) nondetects in affected samples. **NOTE:** It is important to note in the letter text the cause of field duplicate imprecision (i.e., noncompliant %RPD or noncompliant difference between sample and duplicate results). Furthermore, laboratory duplicate data qualifications, as per Brown & Root Environmental convention, shall be matrix-specific but otherwise "across-the-board" for TAL inorganic analyses. However, field duplicate data validation qualifications shall be limited to the field duplicate pair only.

3.1.3.10 Laboratory Control Sample (LCS) Results

Review Laboratory Control Sample Form 7 and associated raw data. Verify that an LCS was analyzed for each matrix and for each batch of twenty samples or batch of samples digested (whichever is more frequent) within an SDG. The quality control criteria established for evaluation of aqueous LCS analyses are 80-120%. NOTE: An aqueous LCS is not required for mercury and cyanide analysis, and silver and antimony are not subject to quality control criteria. Verify that all solid "found values" fall within the EPA established control limits for soils.

Actions - Aqueous LCS: In instances where aqueous LCS %R <80%, qualify as estimated (J) positive results and (UJ) nondetects. If aqueous LCS %R >120, qualify as estimated (J) positive results. Solid LCS: In instances where solid found value is below lower quality control limit, qualify as estimated (J) positive results and (UJ) nondetects. If solid LCS found value exceeds EPA upper limit for soils, qualify as estimated (J) positive results.

3.1.3.11 Method of Standard Additions (MSA)

Review MSA Form 8 and verify instrument linearity by checking that all calibration correlation coefficients (r) are greater than or equal to 0.995. MSAs for a particular analyte in a particular sample may be run more than once. Check reanalyses in instances where initial MSA analysis yields (r) <0.995. It is good practice to review one or two GFAA post-digestion spike (PDS) %Rs via reviewing unspiked and spiked sample concentrations and associated PDS recovery to verify that the Furnace Atomic Absorption Analysis Scheme has been followed as per directional guidance noted on page E-28, document ILM03.0.

Actions - If calibration correlation coefficient (r) <0.995, qualify as estimated (J) positive result and/ or (UJ) nondetect in affected sample. NOTE: The "Q" column on the Form 1 of the affected sample should contain an "S" flag for that particular analyte to indicate that the result was obtained using MSA. A "+" flag should also be recorded when the MSA correlation coefficient (r) <0.995. Review the appropriate Form I and amend if necessary.

3.1.3.12 ICP Serial Dilution Analysis

Review ICP Serial Dilutions Form 9 and associated raw data. Verify that a serial dilution was performed for each matrix and that all ICP analytes are included on the Form 9 with corresponding recovery calculations. Check the calculated Percent Difference (%D) column in instances where the diluted sample result is nondetected. In this situation, the laboratory should report a %D of 100% and not simply list the %D as noncalculable. Overlooking this minor point may result in incomplete sample data qualification in some instances. Amend the Form 9 if necessary. All %Ds for ICP serial dilution analyses

should be <10% when concentrations of corresponding analytes in the original (undiluted) sample are minimally a factor of 50X IDL.

Actions - If %D >10% for an analyte, and the corresponding sample concentration is >50x IDL, qualify as estimated (J) positive results for that analyte in all samples of the same matrix. NOTE: The possibility of negative interference exists when the ICP serial dilution %D >10% and the diluted sample result is significantly > original (undiluted) sample result. Qualify as estimated (J) positive results and (UJ) nondetects in such instances.

3.1.3.13 EPA Analysis Run Logs Form 14s

The Form 14 serves several useful functions. It can be used to obtain sample analysis dates as noted in the heading of the page. Secondly, it is used to record any dilutions as applicable to ICP, GFAA, mercury, and cyanide analyses. And finally, it can be used to verify that GFAA PDS percent recoveries are within the 85-115% quality control limits. Additionally, the data reviewer should be careful to note that one and only one "X" flag has been used to indicate each reported field sample result or quality control sample result; this can be an area of frequent laboratory error.

Actions - If the PDS %R is <85%, qualify as estimated (J) the corresponding positive result and/or (UJ) nondetect in affected sample. If the PDS %R is >115%, qualify as estimated (J) the corresponding positive result in the affected sample; nondetects are not qualified based on high PDS %R.

3.1.3.14 Further GFAA Evaluations

It is necessary to review the raw data for GFAA analyses and verify that all Coefficients of Variation or Relative Standard Deviations (%RSDs) are <20% for reported sample results which exceed the CRDL.

Actions - If the CV or %RSD exceeds 20% and the reported sample result is > CRDL, qualify as estimated (J) positive result in affected sample.

3.1.4 Deliverables Guidance

In addition to any specific USEPA Regional requirements (e.g. data validation memorandum, data summary spreadsheets, USEPA Regional worksheets), all laboratory data package quality control summary forms, sample Form I reports, method blank Form Is, and the Chain-of-Custody report must be given to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct (in accordance with the appropriate USEPA Regional or client requirements) and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

4.0 NON-CLP INORGANICS FOR SOLID AND AQUEOUS MATRICES

4.1 Inorganics (SW-846 6010/7470/9010)

Inductively Coupled Plasma Emission Spectroscopy (ICP) - Analytes commonly analyzed using ICP include: aluminum, barium, beryllium, cadmium, calcium, chromium, cobalt, copper, iron, magnesium, manganese, nickel, potassium, silver, sodium, vanadium, and zinc.

Graphite Furnace Atomic Absorption Spectroscopy (GFAA) - Analytes commonly analyzed using GFAA include: antimony, arsenic, lead, selenium, and thallium.

Cold Vapor Methodology - Mercury is commonly analyzed using cold vapor methodology.

Automated Colorimetric Technique - Cyanide is commonly analyzed using automated colorimetric methodology.

4.1.1 Applicability

These methods are applicable to a large number of matrices including EP extracts, TCLP extracts, industrial wastes, soils, groundwater, aqueous samples, sludges, sediments, and other solid wastes. All matrices require digestion prior to analysis.

Detection limits for analytes are established on a quarterly basis and are both laboratory and instrument specific.

4.1.2 Data Overview Prior to Validation Process

4.1.2.1 Data Completeness

The data reviewer must initially verify that all forms are present and complete (i.e., Forms 1 through 14 must be provided). Areas of special attention when accounting for required forms will include:

- Verify at least one Initial and Continuing Calibration Verification (ICV/CCV) Percent Recovery (%R) calculation as noted on the Calibration Summary (Form 2A or equivalent).
- Verify that a matrix-specific laboratory generated preparation blank has been analyzed for each respective matrix as noted on the blank summary (Form 3 or equivalent) (note, filtered and unfiltered aqueous matrices are to be treated as distinctly different matrices).
- Verify that all ICP analytes are present in both ICSA and ICSAB solutions. Also, verify from the raw data that the laboratory reported all analytes present in solution A to the nearest whole number. It is not uncommon for laboratories to incorrectly report "zeros" or simply leave blank the appropriate solution A columns.
- Check that one matrix spike was analyzed for each particular matrix per analytical batch. Laboratories typically will not include an aqueous matrix for waters if the only aqueous samples contained in the SDG are field quality control blanks (i.e., equipment rinsate blanks and/or field blanks). This is generally accepted without data validation letter text comment. Additionally, the data reviewer may want to verify spiking levels.

- Verify that laboratory duplicate analyses were performed for each matrix. **NOTE**: Field quality control blanks are never to be designated for quality control analyses.

- Check that one Laboratory Control Sample (LCS) was analyzed for each batch of samples per matrix within an SDG. **NOTE**: An aqueous LCS is not required for mercury and cyanide analysis.

- The Method of Standard Additions (MSA) (Form 8 or equivalent) may or may not be present as dictated by Post Digestion Spike (PDS) %Rs. See Section 4.1.3.11 for further details.

- Verify that at least one ICP serial dilution analysis was performed for each matrix within an SDG. **NOTE**: Typically one serial dilution will serve to monitor a given set of samples within an SDG. However, special contractual requirements may necessitate one serial dilution analysis per sample. Ascertain atypical serial dilution frequency requirements through the project manager.

- Simply check that the Form 11 ICP Interelement Correction Factors (Annually) is present.

- Verify that all ICP analytical results fall within the ICP Quarterly Linear Ranges provided on the Form 12 (or equivalent). Verify that no GFAA analytical results exceed the highest standard in the associated GFAA calibration.

- Verify that the Preparation Log accounts for aqueous/soil ICP, AA, mercury, and cyanide digestions/distillation as applicable.

- Examine the Form 14s (or equivalent) to verify that one and only one "X" flag has been used to signify each reported field sample result or quality control sample result. Laboratories are often careless when entering the "X" flag. The validator must verify reported results in instances of discrepancies, amend appropriate forms, and mention in letter text.

Actions - Notify the appropriate laboratory contact of required resubmittals when discrepancies are noted on the forms discussed above.

4.1.3 Technical Evaluation Summary

All data evaluations must be conducted in accordance with current and applicable USEPA Regional protocols and/or specific client contractual requirements and obligations. The applicable documents must be referenced to during the data evaluation process as this Standard Operating Procedure (S.O.P) is intended as proprietary in-house guidance for general inorganic validation practices only.

General parameters such as Data Completeness, Overall System Performance, and Detection Limits must be evaluated concurrently with the parameters discussed below.

4.1.3.1 Holding Times

Holding times are calculated from date of sample collection to date of sample analysis. The date of sample collection must be obtained from the Chain-of-Custody (COC) form. The date of sample analysis is best retrieved from the raw data but may also be obtained from the Form 14.

Sample preservation and holding time requirements are as follows:

- Metals - 6 months; pH <2
- Mercury - 28 days; pH <2
- Cyanide - 14 days; pH >12

Preservation requirements as noted above are applicable to aqueous samples only. Solid samples do not receive preservative but require maintenance at 4°C (±2°C) during shipment and storage.

The above holding times do not apply to leachate analyses. It is suggested that the data reviewer reference SW-846 Method 1311 for any questions regarding TCLP quality control requirements and analytical procedural requirements; these vary significantly from non-TCLP analyses.

Actions - Holding time exceedances result in potentially low-biased results; thus, positive results and nondetects shall be qualified as estimated, (J) and (UJ), respectively. **NOTE**: Gross holding time noncompliances are defined as holding times which are exceeded by a factor or 2X. In these extreme cases, it is practice to reject (R) nondetects while positive results are qualified based upon professional judgment regarding the reliability of the associated data.

4.1.3.2 Initial Calibration Requirements

Calibration must be initiated daily and prior to sample analysis. The following calibration standard requirements must be verified:

- **ICP analyses** - must employ a blank and at least one standard
- **GFAA analyses** - must employ a blank and at least three standards. Additionally, the calibration correlation coefficient (r) must be checked for linearity for each GFAA analysis performed (i.e. r = 0.995 or greater)
- **Mercury analyses** - must employ a blank and at least three standards (r = 0.995 or greater).
- **Cyanide analyses** - must employ a blank and at least three standards (r = 0.995 or greater). **NOTE:** At least two additional standards (a high or low) must be distilled and compared to similar values on the curve. Values of distilled standards should agree within ±10% of undistilled standards.

4.1.3.3 Initial and Continuing Calibration Verification (ICV/CCV)

The ICV/CCV %R quality control limits are 90-110% for ICP metals, 80-120% for GFAA metals and mercury, and 85-115% for cyanide.

Actions - If ICV/CCV %Rs are low, qualify as estimated, (J) positive results and (UJ) nondetects. If ICV/CCV %Rs are high, qualify as estimated (J) positive results; nondetects remain unaffected. **NOTE:** Qualify results of only those samples associated with the noncompliant ICV or CCV (generally, those samples immediately preceding or following the noncompliant standard until the nearest in-control standard).

4.1.3.4 Laboratory Method and Field Quality Control Blanks

Verify that a preparation blank was analyzed for each matrix and for each batch of 20 samples or each sample batch digested, whichever is more frequent. Continuing Calibration Blanks (CCBs) must be run at a frequency of 10% or every 2 hours which ever is more frequent.

The data reviewer will select the maximum contaminant level for each analyte in a particular matrix from which shall be calculated an "action level." The action level shall be established as 5X the maximum contaminant level but must be adjusted for dilution factor, moisture content, and sample weight prior to application.

ICB/CCB contamination shall be applied to all samples within an SDG. Preparation blank contamination shall be applied to samples of the same matrix only. Common practice shall be to qualify as nondetected (U) any contaminant present in a sample which is considered a laboratory artifact (i.e., < the established action level). Professional judgment must be employed when discerning the validity of a concentration present in a field quality control blank. In many instances, contamination present in these blanks can be attributable to "dirty" laboratory practice and not actual field contaminant conditions.

Negative concentrations detected in the laboratory method blanks are indicative of instrumental problems and base-line drifting. Generally, any negative concentration > IDL shall warrant estimation [(J) positives and (UJ) nondetects] of the associated sample data regardless of matrix. Action levels shall not be established for negative concentration levels.

Actions - Qualify as nondetected (U) any positive result within the action level. Qualify as estimated (J) positive results and (UJ) nondetects for analytes for which negative concentrations were noted in the laboratory method blanks (i.e., ICBs, CCBs, and/or preparation blanks).

4.1.3.5 ICP Interference Check Sample Results

Verify that all recoveries for the ICP ICS solution fall within the 80-120% quality control window established for the ICS AB solution.

Actions - For ICS %Rs <80%, qualify as estimated (J) positive results and (UJ) nondetects in affected samples. For ICS %Rs >120%, qualify as estimated (J) positive results in affected samples; nondetects are unaffected by high ICS solution AB recovery. **NOTE:** Affected samples include all samples analyzed between the initial and final solutions or within the eight hour working shift whichever occurs more frequently) which contain Al, Ca, Fe, or Mg at levels >50% of the respective concentration of Al, Ca, Fe, or Mg in the ICS True Solution A.

Next, review concentrations of the four common interfering analytes (aluminum, calcium, iron, and magnesium) in the environmental samples. Any aforementioned interferant present in the environmental samples at concentrations which exceed those present in the ICS solution for that same analyte will require calculation of estimated elemental interference stemming from high interfering analyte concentration. If the previous condition is met; review the ICP/ICS Form 4 or equivalent and note any analytes present in the ICS solution A at levels which exceed the IDL and which are not present in the ICS True solution A. Positive results in the ICS solution A indicate potentially elevated results for this analyte in the affected sample, while negative results in the ICS solution A indicate potentially suppressed results for this analyte in the affected sample.

Next, an estimated elemental interference must be calculated for each analyte > IDL present in the ICS solution A which is not present in the ICS True solution A. The following equation shall be employed:

$$\text{Estimated elemental intf.} = \frac{[\text{Conc. affected analyte in ICS Soln A}] \times [\text{Interferent}]\ [\text{Conc. Sample}]}{\text{Interferent Conc. in ICS Soln A}}$$

It is advisable, although not necessary, to routinely choose the lowest concentration for the interferant level in the ICS so as to calculate the highest estimated interference possible. This method lends itself to a more conservative overall data quality review.

Estimated interferences for each affected analyte > IDL in the ICSA solution must now be compared to the reported environmental sample result for that particular analyte.

Actions - For estimated interferences <10% of the reported sample concentration for a particular affected analyte, take no action; interference is considered negligible. For estimated interferences >10% of the reported sample concentration for a particular affected analyte, qualify (J) positive result and/or (UJ) nondetect for affected analyte in affected sample. (**NOTE**: Calculation of an estimated positive (potentially elevated) interference will have no effect on a reported nondetect; thus, no action is necessary).

4.1.3.6 Matrix Spike Sample Analysis (Pre-digestion)

Verify that at least one matrix spike was performed for each matrix for a given set of samples (maximum of 20 samples) within an SDG. **NOTE:** Filtered and unfiltered samples are to be treated as distinctly different sample matrices and qualified accordingly. Any deviations from the referenced method shall be noted and require laboratory contact for correction.

Aqueous and soil Matrix Spike (MS) recoveries must be within the 75-125% quality control window in instances where the initial sample result is <4X amount spiked. If the initial sample result is >4X the amount spiked and the MS %R is noncompliant, no actions shall be taken.

Actions - For MS %Rs <30%, qualify as estimated (J) positive results and reject (R) nondetects in affected samples. For MS %Rs <75% but >30%, qualify as estimated (J) positive results and (UJ) nondetects in affected samples. For MS %Rs >125%, qualify as estimated (J) positive results in affected samples; nondetects are not compromised by high MS recovery; thus, no actions are warranted.

4.1.3.7 Laboratory Duplicate Precision

Verify that one duplicate sample analysis was performed for each group of samples (maximum of 20 samples) of a similar matrix within an SDG. Control criteria used to evaluate the aqueous laboratory duplicates are as follows:

- a control limit of ±20% for relative percent difference when sample and duplicate results are >5X CRDL
- a control limit of ±1X CRDL for the difference between the sample values when sample and/or duplicate results are <5X CRDL

Control criteria used to evaluate solid laboratory duplicates are as follows:

- a control limit of ±35% for relative percent difference when sample and duplicate results are >5X CRDL

- a control limit of ±2X CRDL for the difference between the sample values when sample and/or duplicate results are <5X CRDL

NOTE: Review Duplicate Summary (Form 6 or equivalent) carefully and verify that the laboratory has in fact reported a %RPD of 200% and not simply recorded the %RPD as noncalculable (in instances where the sample result is positive but the duplicate result is nondetect). Overlooking this minor point may result in incomplete sample data qualification in some instances.

Actions - For any situation involving laboratory duplicate imprecision, qualify as estimated (J) positive results and (UJ) nondetects in affected samples. **NOTE:** It is important to note in the letter text the cause of laboratory duplicate imprecision (i.e., noncompliant %RPD or noncompliant difference between sample and duplicate results).

4.1.3.8 Field Duplicate Precision

Field duplicates can be determined via Project Manager informational documents (i.e., sampling logs) or obtained from Chain-of-Custody (COC) forms. Field duplicates are generally identified as samples having identical sample collection times and dates. In instances were field duplicate samples are included with the sample data set, the following control criteria are generally used to evaluate aqueous field duplicates:

- a control limit of ±30% for relative percent difference when sample and duplicate results are >5X CRDL
- a control limit of ±2X CRDL for the difference between the sample values when sample and/or duplicate results are <5X CRDL

Similarly, the following control criteria are generally used to evaluate solid field duplicates:

- a control limit of ±50% for relative percent difference when sample and duplicate results are >5X CRDL
- a control limit of ±4X CRDL for the difference between the sample values when sample and/or duplicate results are <5X CRDL

NOTE: The %RPD should reflect a difference of 200% and should not simply be recorded as noncalculable in instances where the sample result is positive but the field duplicate result is nondetect. Overlooking this minor point may result in incomplete sample data qualification in some instances.

Actions - For any situation involving field duplicate imprecision, qualify as estimated (J) positive results and (UJ) nondetects in affected samples. **NOTE:** It is important to note in the letter text the cause of field duplicate imprecision (i.e., noncompliant %RPD or noncompliant difference between sample and duplicate results). Furthermore, field duplicate data qualifications, as per Brown & Root Environmental convention, shall be matrix-specific but otherwise "across-the-board" for TAL inorganic analyses.

4.1.3.9 Laboratory Control Sample Results

Verify that an LCS was analyzed for each matrix and for each batch of twenty samples or batch of samples digested (whichever is more frequent) within an SDG. The quality control criteria established for evaluation of aqueous LCS analyses are 80-120%. NOTE: An aqueous LCS is not required for

mercury and cyanide analysis. Verify that all solid "found values" fall within the EPA established control limits for soils.

Actions - Aqueous LCS: In instances where aqueous LCS %R <80%, qualify as estimated (J) positive results and (UJ) nondetects. If aqueous LCS %R >120, qualify as estimated (J) positive results. Solid LCS: In instances where solid found value is below lower quality control limit, qualify as estimated (J) positive results and (UJ) nondetects. If solid LCS found value exceeds EPA upper limit for soils, qualify as estimated (J) positive results.

4.1.3.10 Method of Standard Additions (MSA)

Review MSA Form 8 or equivalent and verify instrument linearity by checking that all calibration correlation coefficients (r) are greater than or equal to 0.995. MSAs for a particular analyte in a particular sample may be run more than once. Check reanalyses in instances where initial MSA analysis yields (r) <0.995. It is good practice to review one or two GFAA post-digestion spike (PDS) %Rs via reviewing unspiked and spiked sample concentrations and associated PDS recovery to verify that the Furnace Atomic Absorption Analysis Scheme has been followed as per directional guidance in the method.

Actions - If calibration correlation coefficient (r) <0.995, qualify as estimated (J) positive result and/ or (UJ) nondetect in affected sample.

4.1.3.11 ICP Serial Dilution Analysis

Verify that all ICP analytes are included on the Form 9 (or equivalent) with corresponding recovery calculations. Check the calculated Percent Difference (%D) column in instances where the diluted sample result is nondetected. In this situation, the laboratory should report a %D of 100% and not simply list the %D as noncalculable. Overlooking this minor point may result in incomplete sample data qualification in some instances. Amend the Form 9 if necessary. All %Ds for ICP serial dilution analyses should be <10% when concentrations of corresponding analytes in the original (undiluted) sample are minimally a factor of 50X IDL.

Actions - If %D >10% for an analyte, and the corresponding sample concentration is >50 IDL, qualify as estimated (J) positive results for that analyte in all samples of the same matrix. NOTE: The possibility of suppressed results exists when the ICP serial dilution %D >10% and the diluted sample result is significantly > original (undiluted) sample result. Qualify as estimated (J) positive results and (UJ) nondetects in such instances.

4.1.3.12 Analysis Run Logs Form 14

The Form 14 or equivalent serves several useful functions. It can be used to obtain sample analysis dates as noted in the heading of the page. Secondly, it is used to record any dilutions as applicable to ICP, GFAA, mercury, and cyanide analyses. And finally, it can be used to verify GFAA PDS percent recoveries within the 85-115% quality control limits. Additionally, the data reviewer should be careful to note that one and only one "X" flag has been used to indicate each reported sample result or quality control sample result; this can be an area of frequent laboratory error.

Actions - If the PDS %R is <85%, qualify as estimated (J) the corresponding positive result and/or (UJ) nondetect in affected sample. If the PDS %R is >115%, qualify as estimated (J) the corresponding positive result in the affected sample; nondetects are not qualified based on high PDS % R.

4.1.3.13 Further GFAA Evaluations

It is necessary to review the raw data for GFAA analyses and verify that all Coefficients of Variation Relative Standard Deviations (%RSDs) are <20% for reported sample results which exceed the CRDL.

Actions - If the CV or %RSD exceeds 20% and the reported sample result is > CRDL, qualify as estimated (J) positive result in affected sample.

4.1.4 Deliverables Guidance

In addition to any specific USEPA Regional requirements (e.g., data validation memorandum, data summary spreadsheets, USEPA Regional worksheets), all laboratory data package quality control summary forms, sample Form I reports, method blank Form Is, and the Chain-of-Custody report must be given to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct (in accordance with the appropriate USEPA Regional or client requirements) and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

5.0 TOXICITY CHARACTERISTIC LEACHING PROCEDURE

5.1 SW-846 Method 1311

5.1.1 Applicability

Method 1311, the Toxicity Characteristic Leaching Procedure (TCLP), is used to determine the mobility/leaching potential of inorganic and organic contaminants in liquid, solid and multi-phase wastes and identify and characterize the waste as hazardous or nonhazardous. Wastes are extracted using two different methods. One method is used prior to the determination of metals, pesticides, and semivolatiles, while another method, zero headspace extraction (ZHE), is used prior to volatile organic analysis.

5.2 Interferences

Besides interferences noted for the specific analytical procedures and extractions, the primary concern is the loss of volatiles via aeration prior to organic determinations.

5.3 Holding Times

Preservatives are not added to samples before extraction. Samples should be stored at 4°C unless refrigeration results in irreversible physical change to the samples. Teflon-lined septum capped vials should be used for samples for volatile analysis. After extraction and prior to analysis, the pH of a TCLP extract should be adjusted to <2 if metallic concentrations are to be measured. Extracts should be preserved for other analytes according to guidance given in the individual analysis methods.

The following holding times apply to TCLP analyses:

Parameter	From Sample Collection to TCLP Extraction	From TCLP Extraction to Preparative Extraction	From Extraction to Analysis
Volatiles (VOAs)	14	Not applicable	14
Semivolatiles (BNAs)	14	7	40
Mercury (Hg)	28	Not applicable	28
All other Metals	180	Not applicable	180

Chain of Custodies (COCs) and raw data are reviewed to determine if holding times were met for all samples. If holding times were not met, all sample data will be qualified as estimated, (J) and (UJ). Nondetects in affected samples will be qualified as rejected, (R), if the holding time was exceeded by a factor of 2 or more.

5.4 Sample Preparation

The selection of extraction reagents is critical to the efficiency of the leaching potential of inorganic and organic chemicals. The extraction fluids must be prepared at pH 4.93 ±0.05 and 2.88 ±0.05 in order to properly leach contaminants in waste samples.

The determination of sample aliquot size for extraction is specified in Method 1311. The determination of percent solids must also be considered in the preparation stage.

5.5 Quality Control

The data package will be reviewed to ensure that the following TCLP quality control (QC) requirements plus the requirements dictated by the specific analytical method have been met.

5.5.1 Blanks

At a minimum, one TCLP extraction blank should be performed for every 20 extractions that have been conducted in an extraction vessel. TCLP extraction blanks should be subjected to the same analytical equipment and preparation reagents used to extract all associated samples. Contamination observed in extraction blanks should be considered when evaluating the sample data for introduced contamination.

5.5.2 Spikes

A minimum of one matrix spike per analytical batch (maximum 20 samples) must be performed for each waste type (e.g., wastewater treatment sludge, contaminated soil, etc.) unless it is already known that the constituents of the waste exceed regulatory levels. In most cases, matrix spikes should be added at a concentration equivalent to the corresponding regulatory level, following the matrix spike addition guidance provided in the analytical method as a minimum.

Internal calibration quantitation methods (such as the method of standard additions) must be employed for a metallic contaminant when the spike %R is <50% and the concentration does not exceed the regulatory level or when the detected concentration is within 20% of the regulatory level. Associated sample results will be qualified as estimated, (J) and (UJ), if internal calibration quantitation methods were not performed when required.

Inorganic contaminant concentrations for TCLP extracts must be quantitated by the method of standard additions (MSA) if analytical methods are determined to be inadequate. MSA curves should be checked for linearity. Positive sample results will qualified as estimated, (J), if the MSA correlation coefficient is <0.995.

5.5.3 Deliverables Guidance

In addition to any work-request requirements (e.g., data validation memorandum), all laboratory data package quality control summary forms, laboratory summaries of sample results and laboratory method blanks, and COCs must be provided for the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

6.0 POLYCHLORINATED DIOXINS AND FURANS FOR SOLID AND AQUEOUS MATRICES

6.1 CLP/SW-846 Method 8280

6.1.1 Applicability

Method 8280 and CLP SOW DFLM1.1 are applicable for the determination of the tetra-, penta-, hexa-, hepta-, and octachlorinated congeners of dibenzo-p-dioxins (PCDDs) and dibenzofurans (PCDFs) (by Gas Chromatography/Mass Spectrometry (GC/MS) via selective ion monitoring) in chemical wastes including fuel oils, sludges, fly ash, still bottoms, reactor residues, soil, and water.

6.1.2 Dioxin Data Package Deliverable Minimum Requirements

The following information must be present in data package prior to the validation effort:

- Appropriate Chain-of-Custody (COC) Form(s)
- Laboratory Case Narrative documenting any particular analytical anomalies encountered and sample description information (i.e., sample cross-reference identifications)
- Calibration Summaries
- Laboratory Control Sample and Duplicate forms
- Single Control Samples and Method Blank Results
- Matrix Spike/Matrix Spike Duplicates
- Retention Time Marker Solutions
- Internal and Recovery Standard Area Summaries

The appropriate laboratory liaison must be contacted immediately if any of the above items have been omitted from the data package.

6.1.3 Technical Data Evaluation

NOTE: Analysis of a fortified standard and blank may be submitted as evidence of compliant Performance Evaluation (PE) analyses as per region-specific requirements. The fortified standard will contain 2,3,7,8-TCDD at a known quantity while the fortified blank will contain 1,2,3,4-TCDD plus other known interferents. The recovery for 2,3,7,8-TCDD recognition must be within the EPA's 99% confidence interval.

6.1.4 Quality Control

6.1.4.1 Holding Times and Sample Preservation

All samples are to be extracted within 30 days of sample collection, and all subsequent analyses are to be conducted within 45 days from the date of collection. **NOTE**: Data qualification based upon holding time noncompliances is rare due to the minor effect of extended storage time on PCDD/PCDF quantitation resulting from the inherent persistence and known stability of these compounds. However, estimation of associated sample data based on holding time shall be subject to the professional judgment of the data validator.

Sample preservation shall be checked by referencing the appropriate Chain-of-Custody (COC) form(s) and verifying that all samples receiving PCDD/PCDF analysis were cooled to and stored at 4°C.

6.1.4.2 Initial Calibration Verification

Review the average Relative Response Factors (RRFs) for all dioxin congeners by recalculating approximately 10% of the reported RRFs while also verifying proper use of quantitation ions. The following ions are specified for selective ion monitoring for PCDDs and PCDFs:

Analyte		Quantitation Ion	Confirmation Ions
PCDDs	Tetra	322	320
	Penta	356	354; 358
	Hexa	390	388; 392
	Hepta	424	422; 426
	Octa	460	458
PCDFs	Tetra	306	304
	Penta	340	338; 342
	Hexa	374	372; 376
	Hepta	408	406; 410
	Octa	444	442

Internal Standards

Analyte	Quantitation Ion	Confirmation Ion
13C12-2,3,7,8-TCDD	334	332
13C12-1,2,3,6,7,8-H_xCDD	404	402
13C12-OCDD	472	470
13C12-2,3,7,8-TCDF	318	316
13C12-1,2,3,4,6,7,8-H_pCDF	420	422

Recovery Standards

Analyte	Quantitation Ion	Confirmation Ion
13C12-1,2,3,4-TCDD	334	332
13C12-1,2,3,7,8,9-H_xCDD	404	402

Next verify the acceptability of isotopic ratios as outlined in the following table:

Analyte		Selected Ions	Relative m/z
PCDDs	Tetra	320/322	0.65-0.89
	Penta	356/358	1.24-1.86
	Hexa	390/392	1.05-1.43
	Hepta	424/426	0.88-1.20
	Octa	458/460	0.76-1.02
PCDFs	Tetra	304/306	0.65-0.89
	Penta	340/342	1.24-1.86
	Hexa	374/376	1.05-1.43
	Hepta	408/410	0.88-1.20
	Octa	442/444	0.76-1.02

Internal Standards

Analyte	Selected Ions	Relative m/z
13C12-2,3,7,8-TCDD	332/334	0.65-0.89
13C12-1,2,3,6,7,8-H_xCDD	402/404	1.05-1.43
13C12-OCDD	470/472	0.76-1.02
13C12-2,3,7,8-TCDF	316/318	0.65-0.89
13C12-1,2,3,4,6,7,8-H_pCDF	420/422	0.88-1.20

Recovery Standards

Analyte	Selected Ions	Relative m/z
13C12-1,2,3,4-TCDD	332/334	0.65-0.89
13C12-1,2,3,7,8,9-H_xCDD	402/404	1.05-1.43

Typically, the data reviewer can expect to associate the following congeners with their associated internal standards as follows:

Internal Standard #1 (13C12-2,3,7,8-TCDD)	TCDD, PeCDD
Internal Standard #2 (13C12-1,2,3,6,7,8-HxCDD)	HxCDD, HpCDD
Internal Standard #3 (13C-OCDD)	OCDD, OCDF
Internal Standard #4 (13C12-TCDF)	TCDF, PeCDF
Internal Standard #5 (13C12-HpCDF)	HxCDF, HpCDF

Additionally, verify that the Relative Standard Deviation (%RSD) for all target compounds and internal standards is ≤15%.

Actions - Qualify as estimated, (J) positive results and (UJ) nondetects in affected samples if RSD is >15%.

Window Defining Mix

This is a retention time check which must be run prior to the continuing calibration. The composition of the window defining mix may or may not be known. Review the following criteria:

- Peak separation must be ≤25% valley criterion for TCDD isomers
- Peak separation must be ≤ the 50% valley criterion for HxCDD isomers
- Multiple ion detection mass chromatograms and reconstructed ion chromatograms should be present for the window defining mix

Actions - Professional judgment (weighted primarily upon chromatographic expertise) must be employed when assigning data qualifications.

6.1.4.3 Continuing Calibration Verification

Evaluation of the CCV involves evaluating the Daily Standard (which is a standard that contains the required target compounds plus internal standards), versus the initial standard.

Verify that a Continuing Calibration Verification (CCV) was analyzed prior to sample analysis and at the beginning of each subsequent 12-hour period. A CCV must also be analyzed at the end of the final analysis period.

The Signal-to-Noise ratio (S/N) for all internal standards must be >10:1. No quality control criteria exist to govern internal standard recovery; however, internal standard advisory recovery limits of 40-120% were established in earlier EPA validation protocol.

Verify that the internal standard area count in the sample is -50% to +100% of the internal standard area count in the associated daily standard.

Complete one Percent Recovery ($\%R_{is}$) calculation for an internal standard as outlined in **equation A** below:

Equation A:

$$\%R_{is} = \frac{(A_{is})\ (Q_{rs})}{(A_{rs})\ (RRF_{is})\ (Q_{is})} \times 100$$

where:

A_{is}	=	area of the quantitation ion of the internal standard
A_{rs}	=	area of the quantitation ion of the recovery standard
Q_{is}	=	ng of internal standard
Q_{rs}	=	ng of recovery standard
RRF_{is}	=	Relative Response Factor for the internal standard as determined from the associated continuing calibration

An RRF shall be calculated for each congener in the CCV solution. A Percent Difference (%D) of 30% from the average RRF must be accomplished for the CCV. **NOTE**: Recalculate some (approximately 10%) of the continuing calibration RRFs for thoroughness.

Actions - Qualify associated sample data as estimated, i.e., (J) positive results and (UJ) nondetects in affected samples in instances where CCV %D >30%. Qualify as rejected (R) all associated sample data in instances where the internal standard S/N ratio <10:1.

6.1.4.4 Laboratory Method Blank Evaluations

Verify that a laboratory generated method blank was analyzed prior to sample analysis and for each matrix and extraction batch for all samples within an SDG. The laboratory method blanks should be free from contamination and/or interferences stemming from glassware involved in sample preparation and subsequent analytical procedures, associated reagents and solvents, etc. The following criteria shall be employed for evaluation of contaminant levels present in laboratory method blanks:

- The signal of any confirmed analyte present in a method blank must be <2% of the signal of the associated internal standard (based on peak height or peak area). Comparison of contaminants present in the blanks at levels below the calibration range (i.e., contaminants present at levels which constitute <2% of the respective internal standard) shall not require reanalyses as stipulated by the method.
- An action level of 5X the maximum contaminant level shall be used in instances of positive detections.
- The data reviewer should complete a detection limit verification calculation.
- Detection limits are sample-specific dependent upon the concentration of a given analyte to produce a signal with a peak height ≥2.5 X the background signal.
- The data reviewer shall consider all applicable sample weight, moisture content, and dilution factors prior to application of the aforementioned action level.
- The data reviewer shall recalculate at least one Detection Limit (DL) using **equation B** as follows:

Equation B:

$$DL = \frac{(2.5)\ (H_x)\ (Q_{is})}{(A_{is})\ (RRF_A)\ (W)}$$

where:

A_{is}	=	area of the quantitation ion of the internal standard
Q_{is}	=	ng of internal standard
H_x	=	peak height of noise for the analyte's quantitation ion
RRF_{is}	=	Relative Response Factor for the analyte as determined from the associated continuing calibration
W	=	dry weight of the sample (g)

Actions - Effects on sample data and subsequent data qualifications shall be upon the professional judgment of the data reviewer, but the following general qualifying guidance shall be employed; Qualify as nondetected (U) any positive result less than the corresponding action level.

6.1.4.5 Duplicate Control Samples

The Duplicate Control Sample (DCS) is a well-characterized matrix which is spiked and analyzed at approximately 10% of the sample load in order to establish method-specific quality control limits. The DCS spike recovery quality control limits of 60-140% shall be employed. Additionally, the RPDs between control sample and duplicate shall be below 50%.

Actions - Qualify as estimated (J) positive results in affected samples when DCS spike recoveries are >140%. Qualify as estimated (J) positive results and (UJ) nondetects in affected samples when DCS spike recoveries are <60%. Qualify as estimated (J) positive results and (UJ) nondetects in affected samples when %RPD between control and duplicate sample exceeds 50%.

6.1.4.6 Matrix Spike/Matrix Spike Duplicate Review

Verify that a matrix spike has been analyzed for each matrix and batch of samples within an SDG.

Verify that the %RSD between matrix spike and duplicate injections is ≤50%. Additionally, the following recovery limits shall be employed for the respective congeners:

Congener	Recovery Limits
TCDD	50-150%
PCDD	50-150%
HxCDD	50-150%
HpCDD	50-150%
OCDD	50-150%
TCDF	50-150%
PeCDF	50-150%
HxCDF	50-150%
HpCDF	50-150%
OCDF	50-150%

Actions - Qualify as estimated (J) only positive results in affected samples when the recovery exceeds the upper quality control limit. Qualify as estimated, (J) positive results and (UJ) nondetects in affected samples when the recovery is below the lower quality control limit.

6.1.4.7 Chromatographic Performance and Evaluation

Verify that the recovery standard area counts are within -50% to +100% of the area counts in the respective daily check standard.

Examine chromatographic acceptability by checking the chromatographic base-line for fluctuation (i.e., raising or lowering), peak shape and resolution. Proper peak resolution between 13C-2,3,7,8-TCDD and

13C-1,2,3,4-TCDD (or 13C-2,3,7,8-TCDD and its closest eluting isomer), shall be attained at a threshold acceptability level of <25%.

Actions - Data qualification shall be based upon the professional judgment of the data reviewer.

6.1.4.8 Sample Quantitation

Confirm the quantitation of at least one Estimated Maximum Positive Concentration (EMPC). The laboratory will report an EMPC as opposed to a confirmed, definite positive hit in instances where the S/N ≥2.5 for both the quantitation ion and confirmation ion for a given target isomer/analyte. The following equation shall be used to verify at least one EMPC calculation:

$$EMPC = \frac{(A_x)\ (Q_{is})}{(A_{is})\ (RRF_A)\ (W)}$$

where: A_x = area of the quantitation or confirmation ion, whichever is lower
Q_{is}, A_{is}, RRF_A, and W are defined in the previous equation.

The data reviewer will also confirm at least one positive detection using the following equation:

$$C_A = \frac{(A_A)\ (Q_{is})}{(A_{is})\ (RRF_A)\ (W)}$$

where: A_{is}, Q_{is}, RRF_A, and W are defined in previous equations
C_A = analyte concentration (ng/g or ug/kg)
A_A = analyte quantitation ion area

NOTE: EMPC values are estimates by definition. If these values are used for risk assessment, it must be understood that an EMPC value is "less certain" that positive results which are qualified (J), since the qualified results meet identification criteria while EMPCs do not.

6.1.5 Deliverables

In addition to any work-request requirements (e.g., data validation memorandum), all laboratory data package quality control summary forms, laboratory summaries of sample results and laboratory method blanks, and COCs must be provided to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct, and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

7.0 MISCELLANEOUS ORGANICS

7.1 Biochemical Oxygen Demand (SM 5210B, EPA 405.1)

7.1.1 Applicability

This method determines oxygen requirements of municipal and industrial wastewaters by measuring the oxygen required for the biochemical degradation of organic material and the oxygen used to oxidize inorganic material such as sulfides and ferrous ion. It may also measure the oxygen used to oxidize forms of nitrogen unless their oxidation is prevented by an inhibitor. Results from this test may be used for the development of engineering criteria for the design of wastewater treatment plants.

7.1.2 Interferences

BOD results can be affected by contamination of the dilution water used in the analysis, the presence of toxicants, or by use of a poor seeding material. Insuring the purity of the dilution water will reduce misleading BOD results. Samples containing toxic substances may require special treatment before analysis.

7.1.3 Holding Times

Samples designated for BOD analysis are collected in high-density polyethylene bottles and stored at 4°C until analysis (unless samples are analyzed within 2 hours of collection). A 24 hour holding time is recommended for this method, however, the maximum holding time for BOD analyzed via EPA method 405.1 is 48 hours. If the holding time is exceeded the sample results are qualified as estimated, (J) and (UJ). Gross exceedance (>2X holding time) may warrant the rejection, (R), of nondetects.

7.1.4 Quality Control

7.1.4.1 Blanks

At a minimum, one laboratory method blank should be analyzed per sample batch (maximum 20 samples). The COCs should be consulted to determine if any field quality control blanks (field, rinsate, equipment, etc.) are associated with the samples. If contamination is noted in the associated blanks, positive sample results < the maximum amount detected in the blanks will be qualified as undetected, (U).

7.1.4.2 Glucose-glutamic Acid Check Standard

BOD is determined from a check standard containing a seed, and a glucose-glutamic acid solution. If the BOD value for this check standard is outside the range of 200 ±37 mg/L, BOD determinations made with the seed and diluted water are rejected.

7.1.5 Deliverables Guidance

The content and format of the data package generated for BOD analysis may vary significantly depending upon the work request.

In addition to any work-request requirements (e.g., data validation memorandum), all laboratory data package quality control summary forms, laboratory summaries of sample results and laboratory method

blanks, and COCs must be provided to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

7.2 Chemical Oxygen Demand (EPA Method 410.1/410.2)

7.2.1 Applicability

Methods 410.1 and 410.2 determine the quantity of oxygen required to oxidize organic matter in a domestic or industrial waste sample under specific conditions of oxidizing agent, temperature, and time. Method 410.1 is applicable to samples containing an organic carbon concentration greater than 50 mg/L, which Method 410.2 is applied to samples containing an organic carbon concentration in the range of 5 to 50 mg/L.

7.2.2 Interferences

Traces of organic material, rise in temperature, or high concentrations of chloride may cause error in determination of COD. Glassware used in the procedure would be conditioned by running blank procedures to eliminate traces of organic material, and contamination of the distilled water used in the procedure must be avoided. Loss of volatile substances may be minimized by cooling the flask used in the analysis during the addition of the sulfuric acid solution. Positive interferences caused by chlorides are eliminated with the addition of mercuric sulfate.

7.2.3 Holding Times

Glass bottles are recommended for sample collection, although plastic may be permissible if the containers are free of organic material contamination. Samples are preserved with sulfuric acid to a pH <2 and stored at 4°C until analysis. The maximum holding time specified (from sample collection to analysis) is 28 days. (The method does recommend that biologically active samples be tested as soon as possible.) Sample results will be qualified as estimated, (J) and (UJ), if this holding time is exceeded. Gross holding time exceedances (>2X holding time) will warrant rejection (R); of nondetects.

7.2.4 Quality Control

7.2.4.1 Blanks

A low COD water blank must be run simultaneously with the environmental sample to quantitate the amount of COD present in the environmental sample. Additionally, one laboratory method blank should be analyzed per sample batch. The COCs should be consulted to determine if any field quality control blanks (field, rinsate, equipment, etc.) are associated with the samples. If contamination is noted in the associated blanks, positive sample results < the maximum amount detected in the blanks will be qualified as undetected, (U).

7.2.5 Deliverables Guidance

The content and format of the data package generated for COD analysis may vary significantly depending upon the work request.

In addition to any work-request requirements (e.g., data validation memorandum), all laboratory data package quality control summary forms, laboratory summaries of sample results and laboratory method blanks, and COCs must be provided to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

7.3 Total Recoverable Petroleum Hydrocarbons (EPA 418.1)

7.3.1 Applicability

EPA Method 418.1 is used to measure the amount of fluorocarbon-113 extractable petroleum hydrocarbons from aqueous matrices. With modification, solid waste petroleum hydrocarbons can also be measured. Infrared analysis of a waste sample extract is performed by direct comparison with a calibration standard plot.

7.3.2 Interferences

The measurement of petroleum hydrocarbons by infrared analysis is subject to interference. The addition of silica gel to the sample reduces the effects of interference.

7.3.3 Holding Times

Samples are collected in glass bottles. Aqueous samples are preserved with hydrochloric acid to a pH <2 and are cooled to 4°C. Solid samples are stored at 4°C until analysis.

A 28-day holding time is used to evaluate the samples. Chain of Custodies (COCs) and raw data are reviewed to determine if holding times were met for all samples. Samples results will be qualified as estimated, (J) and (UJ), if holding times are exceeded. Gross holding time violations (>2X holding time) will warrant rejection, (R), of nondetects.

7.3.4 Quality Control

Quality control criteria are not specified in Method 418.1. However, if quality control analyses are performed by the laboratory, the following criteria will be used to evaluate the associated data.

7.3.4.1 Calibration

The calibration curve of absorbance versus concentration of petroleum hydrocarbons in known standards should be checked for linearity. Generally, associated sample data are qualified as estimated, (J) and (UJ), if the calibration curve correlation coefficient is <0.995. Professional judgment should be used to qualify sample data in cases when sample results fall outside the linear portion of the calibration curve.

If analyzed, the percent recovery (%R) of a continuing calibration verification (CCV) standard will be evaluated using an 85-115% quality control range. Associated sample data will be qualified as estimated, (J) and (UJ), if the CCV %R is <85%. Only positive results in the affected samples will be qualified as estimated, (J), if the CCV %R is >115%.

7.3.4.2 Blanks

Laboratory method blanks, if analyzed, should be evaluated for contamination. The COCs should be consulted to determine if any field quality control blanks (field, rinsate, equipment, etc.) are associated with the samples. If contamination is noted in any of the associated blanks, positive sample results < the maximum amount detected in the blanks will be qualified as undetected, (U). Sample aliquot and moisture content factors will be taken into consideration when qualifying the associated sample data.

7.3.4.3 Spikes/Duplicates

If a spiked sample is analyzed, a 75-125% quality control range will be used to evaluate the spike %R. Associated sample data will be qualified as estimated, (J) and (UJ), when the spiked sample %R is <75%. When the %R is >125%, only positive results are impacted and qualified as estimated, (J). If a spike %R is <30%, associated nondetects will be qualified as rejected, (R), and positive results will be qualified as estimated, (J).

7.3.4.4 Duplicates

Qualification of sample data based on duplicate precision is left to the professional judgment of the validator. Generally, a ±20% aqueous quality control limit and a ±30% solid quality control limit are used to evaluate the Relative Percent Difference (RPD) between the sample and laboratory duplicate results; a ±30% aqueous quality control limit and a ±50% solid quality control limit are generally used to evaluate the RPD between field duplicate results; qualification based on field duplicate imprecision is applied only to the field duplicate pair for general chemistry parameters.

7.3.4.5 Sample Quantitation

All reported sample concentrations should fall within the range of the calibration curve. If samples containing concentrations > the highest calibration standard were not diluted and reanalyzed, the associated sample data will be qualified as estimated, (J).

Verify that sample results were properly quantitated.

7.3.5 Deliverables Guidance

The content and format of the data package generated for petroleum hydrocarbon analysis may vary significantly depending upon the work request.

In addition to any work-request requirements (e.g., data validation memorandum), all laboratory data package quality control summary forms, laboratory summaries of sample results and laboratory method blanks, and COCs must be provided to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

7.4 Total Organic Carbon (EPA SW846 Method 9060)

7.4.1 Applicability

Method 9060 is used to measure concentrations of total organic carbon (TOC) in excess of 1 mg/L in domestic and industrial wastes, groundwater, and surface and saline waters. The organic carbon is first converted to carbon dioxide: the CO_2 is then measured directly using an infrared detector or converted to methane and measured by a flame ionization detector.

7.4.2 Interferences

The presence of inorganic carbon (i.e., carbonate and bicarbonate) must be considered. These substances can be accounted for in the sample calculation or eliminated by acidification and degassing before analysis. If degassing is utilized, volatilization of organic carbon can occur.

7.4.3 Holding Times

Although it is preferable that samples are collected in glass bottles, plastic containers may be used if it is established that the containers do not contribute contaminating organics to the samples. Samples are preserved with hydrochloric or sulfuric acid to a pH <2 and are cooled to 4°C. Protection from light is important for TOC analysis.

Although a precise holding time (elapsed time period from sample collection to analysis) is not stated in the method, a 28-day holding time will be used to evaluate the samples. Chain of Custodies (COCs) and raw data are reviewed to determine if holding times were met for all samples. Samples results will be qualified as estimated, (J) and (UJ), if holding times are exceeded. Gross holding time violations (>2X holding time) will warrant rejection, (R), of nondetects.

7.4.4 Quality Control

7.4.4.1 Calibration

A calibration curve should be prepared comparing concentrations of known standards to actual TOC readings. (Samples should be analyzed in quadruplicate; the average value and the range of readings should be reported.) The calibration curve should be checked for linearity. Generally, associated sample data are qualified as estimated, (J) and (UJ), if the calibration curve correlation coefficient is <0.995. Professional judgment should be used to qualify sample data in cases when sample results fall outside the linear portion of the calibration curve.

The method requires that a continuing calibration verification (CCV) standard be analyzed every 15 samples. An 85-115% quality control range will be used to evaluate the percent recovery (%R) of the CCV. Associated sample data will be qualified as estimated, (J) and (UJ), if the CCV %R is <85%. Only positive results in the affected samples will be qualified as estimated, (J), if the CCV %R is >115%.

7.4.4.2 Blanks

At a minimum, one laboratory method blank (other than the blank used for the calibration curve) should be analyzed per sample batch (maximum of 20 samples). The COCs should be consulted to determine if any field quality control blanks (field, rinsate, equipment, etc.) are associated with the samples. If contamination is noted in the associated blanks, positive sample results < the maximum amount

detected in the blanks will be qualified as undetected, (U). Sample digestion and moisture content factors will be taken into consideration when qualifying the associated sample data.

7.4.4.3 Spikes/Duplicates

A spiked sample and spiked duplicate sample should be analyzed for every 10 samples. If a spike or duplicate spike %R is <75%, the associated sample data will be qualified as estimated, (J) and (UJ). If the %R is >125%, only positive results are impacted and qualified as estimated, (J). If a spike %R is <30%, associated nondetects will be qualified as rejected, (R), and positive results will be qualified as estimated. (J).

Qualification of sample data based on duplicate precision is left to the professional judgment of the validator. Generally, a ±20% aqueous quality control limit and a ±30% solid quality control limit are used to evaluate the relative percent difference (RPD) between the spiked sample and spiked duplicate sample results. A ±30% aqueous control limit and a ±50% solid quality control limit are generally used to evaluate the RPD between field duplicate results; qualification based on field duplicate imprecision is applied only to the field duplicate pair for general chemistry parameters.

7.4.4.4 Sample Quantitation

All reported sample concentrations should fall within the range of the calibration curve. If samples having concentrations > the highest calibration standard were not diluted and reanalyzed, the associated sample data will be qualified as estimated, (J).

Verify that sample results were properly quantitated.

7.4.5 Deliverables Guidance

The content and format of the data package generated for TOC analysis may vary significantly depending upon the work request.

In addition to any work-request requirements (e.g., data validation memorandum), all laboratory data package quality control summary forms, laboratory summaries of sample results and laboratory method blanks, and COCs must be provided to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct, and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

7.5 Total Organic Halides (EPA SW-846 Method 9020B)

7.5.1 Applicability

Method 9020B uses carbon adsorption with a microcoulometric-titration detector to measure the concentration of total organic halides (TOX) as chloride in drinking and ground waters. This method detects all organic halides containing chlorine, bromine, and iodine.

- Organic halides containing fluorine cannot be measured.
- TOX adsorbed to undissolved solids cannot be measured.

- The concentration of inorganic halides in the sample can not exceed the organic halide concentration by a factor >20,000.

7.5.2 Interferences

Interferences from contaminated reagents, glassware, activated carbon, and other laboratory devices must be minimized. Special care must be taken to clean, dry, and store materials used during analysis to protect against contamination from halogenated organic vapors and oily residue.

Suspended matter, which can clog adsorption columns, must be eliminated prior to sample analysis by decanting the aqueous phase or centrifuging to separate undissolved materials.

7.5.3 Holding Times

Samples should be collected in duplicate, preserved with sulfuric acid to a pH <2, and cooled to 4°C. Plastic or glass containers may be used. All samples must be protected from light.

Samples must be analyzed within 28 days of collection. Chain of Custodies (COCs) and raw data are reviewed to determine if holding times were met for all samples. Samples results will be qualified as estimated, (J) and (UJ), if holding times are exceeded. Gross holding time violations (>2X holding time) will warrant rejection (R), of nondetects.

7.5.4 Quality Control

7.5.4.1 Calibration

Pyrolyzation

The following requirements must be met during the pyrolysis stage:

- The adsorption efficiency of the activated carbon must be checked. The percent recovery (%R) of a standard should be within ±10%.
- A nitrate-wash blank must be run after every 10 pyrolyzations.
- Pyrolysis instrument calibration standards, which should be run after every 10 determinations, must be analyzed in duplicate. The %R for these standards must be within ±10% of the true value.

If any of these requirements have not been met, associated sample data will be qualified as estimated, (J) and (UJ).

Microcoulometric Analysis

The method requires that a continuing calibration verification (CCV) standard be analyzed every 15 samples. Although the method does not specify criteria to evaluate the CCV, an 85-115% quality control range will be used for validation purposes. Affected sample data will be qualified as estimated, (J) and (UJ), if the CCV %R is <85%. Only positive results in the affected samples will be qualified as estimated, (J), if the CCV %R is >115%.

7.5.4.2 Blanks

A minimum of two method blanks (other than the blanks used for the calibration and pyrolyzation) should be analyzed to establish the repeatability of the method background and the background should be monitored by analyzing method blanks after every eight samples. The COCs should be consulted to determine if any field quality control blanks (field, rinsate, equipment, etc.) are associated with the samples. If contamination is noted in the associated blanks, positive sample results < the maximum amount detected in the blanks will be qualified as undetected, (U). Dilution factors will be taken into consideration when qualifying the associated sample data. (Nitrate-wash blanks, which are considered to be laboratory method blanks, should not be used to qualify sample results since contamination in these blanks is already accounted for in the sample calculation.)

7.5.4.3 Spikes/Duplicates

A spiked sample should be analyzed between every 10 samples. If a spike or duplicate spike %R is not within 75-125%, the associated sample data will be qualified as estimated, (J) and (UJ). If the %R is >125%, only positive results are impacted and qualified as estimated, (J). If a spike %R is <30%, associated nondetects will be qualified as rejected, (R), and positive results will be qualified as estimated, (J).

The method requires that all samples be seen in duplicate. Qualification of sample data based on duplicate precision is left to the professional judgment of the validator. Generally, a ±20% aqueous quality control limit and a ±30% solid quality control limit are used to evaluate the relative percent difference (RPD) between the sample and laboratory duplicate sample results. A ±30% aqueous quality control limit and a ±50% solid quality control limit are generally used to evaluate the RPD between field duplicate results; qualification based on field duplicate results for general chemistry parameters are applied to the field duplicate pair only.

7.5.4.4 Breakthrough

Check the extent of organohalide breakthrough from the first column. The second column measurement should not exceed 10% of the sum of the measurements from both columns. Positive results will be estimated, (J), if the 10% quality control limit was exceeded.

7.5.4.5 Sample Quantitation

The following equation is used to calculate TOX:

$$TOX\ (\mu g/L) = \frac{(conc_1 - conc_{blank}) + (conc_2 - conc_{blank})}{vol_{sample}}$$

where:

$conc_1$	=	concentration of chloride measured on first column (μg)
$conc_2$	=	concentration of chloride measured on second column (μg)
$conc_{blank}$	=	average, daily concentration in nitrate-wash blanks (μg)
vol	=	volume of sample aliquot (L)

Verify that sample results were accurately quantitated.

7.5.5 Deliverables Guidance

The content and format of the data package generated for TOX analysis may vary significantly depending upon the work request.

In addition to any work-request requirements (e.g., data validation memorandum), all laboratory data package quality control summary forms, laboratory summaries of sample results and laboratory method blanks, and COCs must be provided to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

8.0 MISCELLANEOUS INORGANICS

8.1 Carbonate/Bicarbonate Alkalinity (EPA 600 Series Method 310.2)

8.1.1 Applicability

Method 310.2 is an automated method used to measure alkalinity (as $CaCO_3$) at concentrations ranging from 10 to 200 mg/L in domestic and industrial effluents, and drinking, surface and saline waters.

8.1.2 Interferences

Since the method of analysis is colorimetric, primary interferences for this method include turbidity and color. Samples can be filtered prior to analysis to reduce interferences from turbidity.

8.1.3 Holding Times

Samples should be collected in plastic or glass containers and cooled to 4°C. No preservative is needed.

Holding time is defined as the elapsed time period from sample collection to analysis. The holding time for this method is 14 days. Chain of Custodies (COCs) and raw data are reviewed to determine if holding times were met for all samples. Positive results and nondetects will be qualified as estimated, (J) and (UJ), respectively, if holding times were exceeded. Gross holding time violations (>2X holding time) will warrant rejection, (R), of nondetects.

8.1.4 Quality Control

Quality control analyses and criteria (i.e., calibrations, blanks, spikes, etc.) are not specified in Method 310.2. However, if these analyses were performed by the laboratory, the following criteria will be used to evaluate the associated sample data.

8.1.4.1 Calibration

According to the method, a calibration curve should be prepared by plotting peak heights of standards to known concentrations. This curve should be checked for linearity. Generally, associated sample data are qualified as estimated, (J) and (UJ), if the calibration curve correlation coefficient is <0.995. Professional judgment should be used to qualify sample data in cases when sample results fall outside the linear portion of the calibration curve.

If analyzed, the percent recovery (%R) of a continuing calibration verification (CCV) standard will be evaluated using an 85-115% quality control range. Associated sample data will be qualified as estimated, (J) and (UJ), if the CCV %R is <85%. Only positive results in the affected samples will be qualified as estimated, (J), if the CCV %R is >115%.

8.1.4.2 Blanks

Laboratory method and field quality control blanks, if analyzed, should be identified and assessed for introduced contamination. Field quality control blanks (field, rinsate, equipment, etc.) can be identified by consulting the COCs. If contamination is noted in the associated blanks, positive sample results < the maximum amount detected in the blanks will be qualified as undetected, (U). Sample digestion and moisture content factors will be taken into consideration when qualifying the associated sample data.

8.1.4.3 Spikes

If a spiked sample is analyzed, a 75-125% quality control range will be used to evaluate the spike %R. Associated sample data will be qualified as estimated, (J) and (UJ), when the spiked sample %R is <75%. When the %R is >125%, only positive results are impacted and qualified as estimated, (J). If a spike %R is <30%, associated nondetects will be qualified as rejected, (R), and positive results will be qualified as estimated, (J).

8.1.4.4 Duplicates

Qualification of sample data based on duplicate precision is left to the professional judgment of the validator. Generally, an aqueous quality control limit of ±20% and a solid quality control limit of ±30% is used to evaluate the relative percent difference (RPD) between the sample and laboratory duplicate results. An aqueous quality control limit of ±30% and a solid quality control limit of ±50% are generally used to evaluate the RPD between field duplicate results; qualification based on field duplicate imprecision for general chemistry parameters is applied to the field duplicate pair only.

8.1.4.5 Sample Quantitation

All reported sample concentrations should fall within the range of the calibration curve. If samples having detected concentrations > the highest calibration standard were not diluted and reanalyzed, associated sample data will be qualified as estimated, (J).

The validator should verify that sample results were properly quantitated.

8.1.5 Deliverables Guidance

The content and format of the data package generated for alkalinity analysis may vary significantly depending upon the work request.

In addition to any work-request requirements (e.g., data validation memorandum), all laboratory data package quality control summary forms, laboratory summaries of sample results and laboratory method blanks, and COCs must be provided to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

8.2 Anions (EPA Method 300.0)

The Determination of Inorganic Anions in Water by Ion Chromatography

8.2.1 Applicability

Method 300.0 is a Ion Chromatographic (IC) Procedure used to determine the inorganic anions chloride, fluoride, nitrate (as nitrogen), nitrite (as nitrogen), ortho-phosphate (as phosphorus), and sulfate in drinking water, surface water, and mixed domestic and industrial wastewater.

8.2.2 Interferences

Interferences may be caused by particulates or other substances present in the sample that may have retention times similar to the particular anion of interest. Also, a large concentration of one anion may mask the resolution of an adjacent anion. Sample dilution and/or spiking (to generate a sample-specific calibration) may be employed to resolve these problems. Additionally, method interferences may be caused by contaminants in reagent water, reagents, glassware, and other elements of sample processing.

The fluoride peak, in particular, may be affected by a water dip (a negative peak) that elutes near it. This problem can be eliminated by the addition of 1 mL of concentrated sodium carbonate eluent solution to 100 mL of each standard and sample.

8.2.3 General Laboratory Practices

The laboratory should spike and analyze a minimum of 10% of all samples to monitor continuing laboratory performance. Field and laboratory duplicates should also be analyzed.

Validation: The validator should check the work request to ascertain what contracted quality control analyses are required. Likewise, the validator should check with the project manager to determine which samples (if any) are field duplicates or field quality control blanks.

Before any analyses are performed the laboratory must demonstrate the ability to generate acceptable accuracy and precision using a blank spike sample (laboratory control sample; LCS), which is a reagent water blank spiked with a known concentration of stock standard solutions at the concentrations stipulated in EPA Method 300.0, Sections 8.2.2 through 8.3.1.

Analysis of this blank spike sample will indicate the accuracy of the measurement via the calculation of Percent Recovery (%R). Upper and lower control limits for %Rs should be calculated. These control limits can then be used to construct control charts that may be useful in observing trends in performance. This blank spike sample should also be duplicated and analyzed to indicate precision of the measurements between identical samples through comparison of the recoveries generated via the blank spike and blank spike duplicate analyses. The blank spike/blank spike duplicate analyses should be performed with the same frequency as matrix spike/matrix spike duplicate analyses.

Validation: The data reviewer shall examine The %Rs to determine if they are within the laboratory generated control limits. If %Rs are below the control limits positive results will be qualified (J) and nondetected results will be qualified (UJ). If %Rs are above the control limits only positive results will be qualified (J). If %Rs are extremely low (less than 10%) the laboratory should reanalyze the blank spike and blank spike duplicate samples. If the laboratory does not reanalyze these samples then qualifications are necessary. Positive results will be qualified as estimated (J) and nondetects will be rejected (R), when %Rs are less than 10%.

The reviewer should also examine the Relative Percent Difference (RPD) between the calculated %Rs. If the RPD is above an acceptable level qualify positive results (J) and use professional judgment to determine if nondetects should be qualified (UJ).

8.2.4 Holding Times

The following table indicates sample preservation and holding time requirements:

Analyte	Preservation	Holding time
Chloride	None required	28 days
Fluoride	None require	28 days
Nitrate-N	Cool to 4°C	48 hours
Nitrite-N	Cool to 4°C	48 hours
o-Phosphate-P	Filter & cool to 4°C	48 hours
Sulfate	Cool to 4°C	28 days

Validation: Holding times are calculated from time of collection obtained from Chain-of-Custody (COC) forms to time of analysis. Positive results in samples analyzed past holding times are qualified as estimated (J); nondetects (UJ). If holding times are exceeded by a factor of 2 or more it is considered to be a gross exceedance; positive results are qualified as estimated, (J), and nondetects are rejected, (R). Results are considered to be biased low when holding times are exceeded.

8.2.5 Sample Preparation

Samples containing particles greater than 0.45 microns and reagent solutions containing particles greater than 0.20 microns require filtration to prevent damage to the instrument columns and flow systems.

8.2.6 Calibration and Testing

Per each analyte of interest, calibration standards at a minimum of 3 concentration levels should be prepared (generated from a stock solution and diluted appropriately) and analyzed along with a blank. One of the standard concentrations must be near but above the MDL. A sufficient number of standards should be analyzed to accurately define a calibration curve.

A consistent aliquot (injections of 0.1 to 1.0 mL) for samples and standards must be used. An automated constant volume injection system may be employed.

Calibration for each analyte should be verified daily, or whenever the anion eluent is changed, and after every 20 samples. Retention times must agree within ±10%. If agreement is not met a new calibration curve should be generated for that analyte.

Validation: The validator will evaluate the 3-point calibration and verify that one of the points was at a concentration near the MDL. Next, the retention times will be examined to ensure that they agree within ±10%. If the retention time is outside the ±10% window, the result for the affected analyte will be qualified as estimated; (J) positive results and (UJ) nondetects.

If peak response exceeds the linear calibration range of the instrument, the sample should be diluted with the appropriate amount of reagent water and reanalyzed. If the chromatogram does not produce adequate resolution or if identification of the chromatographic peaks are questionable, the sample should be spiked with the appropriate amount of standard and reanalyzed.

Validation: The validator will review chromatograms to verify the absence of a water dip (see Section 8.2.2) and to verify that peak responses are within the linear range and that adequate resolution was achieved. If any noncompliances exist they should be noted. Qualifications will be made per situation, based upon professional judgment.

8.2.7 Blank Contamination

Method blanks (reagent water) should be analyzed at the beginning of each sample batch (maximum of 20 samples) to ensure that there is no carryover or contamination from glassware and/or reagents.

Validation: Blank results should be reported for each sample data set. If contamination is noted in the blanks, the maximum concentration of each contaminant should be used to set action. Action levels are set using professional judgment based upon comparability of the sample result with concentration of the blank contaminant. Results reported for contaminants found in samples that are greater than the detection limit and within the action level are qualified as undetected, (U). The same process is repeated for field quality control blanks.

8.2.8 Sample Quantitation

A standard curve should be generated by plotting anion peak size in area units against standard anion solution concentration values. Sample concentration can then be calculated by comparing sample peak response with the standard curve. Sample data results should be reported in mg/L.

Validation: The validator shall compare sample results against standard results to confirm that the samples were properly quantitated.

8.2.9 Deliverables Guidance

The content and format of the data package generated for anion analysis may vary significantly dependent upon the work request.

In addition to any work-request requirements (e.g., data validation memorandum, data spread sheet), all laboratory data package quality control summary forms, laboratory summaries of sample data results and method blank analyses and the chain-of-custody report must be given to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

8.3 Bromide (EPA 600 Series Method 320.1)

8.3.1 Applicability

Method 320.1 is a titrimetric method used to determine the concentration of bromide in domestic and industrial effluents, and drinking, surface and saline waters. Bromide concentrations ranging from 2 to 20 mg/L can be measured by this method.

8.3.2 Interferences

Interferences can be caused by the presence of organic matter, iron, and manganese. Pretreatment of samples with calcium oxide removes or reduces these interferences to insignificant concentrations.

Color interferes with the observation of indicator and bromine-water color changes. Steps can be taken during analysis to eliminate this interference (e.g., the use of a pH meter instead of a pH indicator).

8.3.3 Holding Times

Samples should be collected in plastic or glass bottles and cooled to 4°C. No preservative is needed.

Holding time is defined as the elapsed time period from sample collection to analysis. A 28-day holding time is specified for analysis. Chain of Custodies (COCs) and raw data are reviewed to determine if holding times were met for all samples. Positive results and nondetects will be qualified as estimated, (J) and (UJ), respectively, if holding times were exceeded. Gross holding time violations (>2X holding time) will warrant rejection, (R), of nondetects.

8.3.4 Quality Control

Quality control analyses (i.e., blanks, spikes, etc.) are not specified in Method 320.1. However, if these analyses were performed by the laboratory, the following criteria will be used to evaluate the associated sample data.

8.3.4.1 Verification Standard

The percent recovery (%R) of a verification standard, if analyzed, will be evaluated using an 85-115% quality control criteria. Associated sample data will be qualified as estimated, (J) and (UJ), if the %R is <85%. Only positive results in the affected samples will be qualified as estimated, (J), if the %R is >115%.

8.3.4.2 Blanks

Laboratory method and field quality control blanks, if analyzed, should be evaluated for contamination. Field quality control blanks (field, rinsate, equipment, etc.) can be identified by consulting the COCs. If contamination is noted in the associated blanks, positive sample results < the maximum amount detected in the blanks will be qualified as undetected, (U). Sample digestion and moisture content factors will be taken into consideration when qualifying the associated sample data.

8.3.4.3 Spikes

If a spiked sample is analyzed, a 75-125% quality control range will be used to evaluate the spike %R. Associated sample data will be qualified as estimated, (J) and (UJ), when the spiked sample %R is <75%. When the %R is >125%, only positive results are impacted and qualified as estimated, (J). If a spike %R is <30%, associated nondetects will be qualified as rejected, (R), and positive results will be qualified as estimated, (J).

8.3.4.4 Duplicates

Qualification of sample data based on duplicate precision is left to the professional judgment of the validator. Generally, an aqueous quality control limit of ±20% and a solid quality control limit of ±30% is used to evaluate the relative percent difference (RPD) between the sample and laboratory duplicate results. An aqueous quality control limit of ±30% and a solid quality control limit of ±50% are generally used to evaluate the RPD between field duplicate results; qualification based on field duplicate imprecision for general chemistry parameters is applied to the field duplicate pair only.

8.3.4.5 Sample Quantitation

All reported sample concentrations should fall within the range of 2 to 20 mg/L. If samples having detected concentrations >20 mg/L were not diluted and reanalyzed, the associated sample data will be qualified as estimated, (J).

The validator should verify that sample results were properly quantitated.

8.3.5 Deliverables Guidance

The content and format of the data package generated for bromide analysis may vary significantly depending upon the work request.

In addition to any work-request requirements (e.g., data validation memorandum), all laboratory data package quality control summary forms, laboratory summaries of sample results and laboratory method blanks, and COCs must be provided to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

8.4 Fluoride (EPA 600 Series Method 340.2)

8.4.1 Applicability

Method 340.2 is a potentiometric method which uses an ion selective electrode to measure concentrations of fluoride in domestic and industrial effluents, and drinking, surface and saline waters. The practical range of determination is 0.1 to 1,000 mg/L.

8.4.2 Interferences

The pH of samples can cause significant interferences. The ideal pH range of a sample is between 5 and 9.

Complexing cations, such as Si^{+4}, Fe^{+3}, and Al^{+3}, can produce additional interferences during fluoride determinations. Samples can be treated with a pH 5.0 buffer containing a strong chelating agent to eliminate these interferences.

8.4.3 Holding Times

Samples are to be collected in plastic bottles. No preservative is required.

A 28-day holding time (elapsed time period from sample collection to analysis) is specified for analysis. Chain of Custodies (COCs) and raw data are reviewed to determine if holding times were met for all samples. Sample data will be qualified as estimated, (J) and (UJ), if holding times were exceeded. Gross holding time violations (>2X holding time) will warrant rejection, (R), of nondetects.

8.4.4 Quality Control

Quality control analyses (i.e., calibrations, blanks, spikes, etc.) are not specified in Method 340.2. However, if these analyses were performed by the laboratory, the following criteria will be used to evaluate the associated sample data.

8.4.4.1 Calibration

According to the method, the calibration curve should consist of standards ranging in concentration from 0 to 2 mg/L. Semi-logarithmic graph paper should be used to plot the known concentration of the standard versus the electrode potential.

If a continuing calibration verification (CCV) standard is analyzed, the percent recovery of the standard will be evaluated using 85-115% quality control limits. Associated sample data will be qualified as estimated, (J) and (UJ), if the CCV %R is <85%. Only positive results in the affected samples will be qualified as estimated, (J), if the CCV %R is >115%.

8.4.4.2 Blanks

Laboratory method and field quality control blanks, if analyzed, should be assessed for introduced contamination. Field quality control blanks (field, rinsate, equipment, etc.) can be identified by consulting the COCs. If contamination is noted in the associated blanks, positive sample results < the maximum amount detected in the blanks will be qualified as undetected, (U). Sample digestion and moisture content factors will be taken into consideration when qualifying the associated sample data.

8.4.4.3 Spikes

If a spiked sample is analyzed, a 75-125% quality control range will be used to evaluate the spike %R. Associated sample data will be qualified as estimated, (J) and (UJ), when the spiked sample %R is <75%. If the %R is >125%, only positive results are impacted and qualified as estimated, (J). If a spike %R is <30%, associated nondetects will be qualified as rejected, (R), and positive results will be qualified as estimated, (J).

8.4.4.4 Duplicates

Qualification of sample data based on duplicate precision is left to the professional judgment of the validator. Generally, an aqueous quality control limit of ±20% and a solid quality control limit of ±30% is used to evaluate the relative percent difference (RPD) between the sample and laboratory duplicate results. An aqueous quality control limit of ±30% and a solid quality control limit of ±50% is used to evaluate the RPD between field duplicate results; qualification based on field duplicate imprecision for general chemistry parameters is applied to the field duplicate pair only.

8.4.4.5 Sample Quantitation

All reported sample concentrations should fall in the range of the calibration curve. If samples having detected concentrations > the highest calibration standard were not diluted and reanalyzed, associated sample data will be qualified as estimated, (J).

The validator should verify that sample results were properly quantitated.

8.4.5 Deliverables Guidance

The content and format of the data package generated for fluoride analysis may vary significantly depending upon the work request.

In addition to any work-request requirements (e.g., data validation memorandum), all laboratory data package quality control summary forms, laboratory summaries of sample results and laboratory method blanks, and COCs must be provided to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

8.5 Nitrogen (Various)

8.5.1 Nitrate-Nitrogen (EPA 300 Series Method 352.1)

8.5.1.1 Applicability

Method 352.1 is a brucine, colorimetric method used to measure nitrate-nitrogen at concentrations ranging from 0.1 to 2 mg/L in domestic and industrial effluents, and drinking, surface and saline waters.

8.5.1.2 Interferences

The following is a list of interferences observed for this method:

- Uniform temperature control is extremely critical during the color development stage. Erratic heating can produce inconsistent results.
- Strong oxidizing or reducing agents, residual chloride, ferrous and ferric iron, quadrivalent manganese, and salinity in samples can create interferences.
- Interferences from naturally colored samples and dissolved organic matter can affect color during heating and produce erroneous results.

8.5.1.3 Holding Times

Samples should be collected in plastic or glass containers and cooled to 4°C.

The holding time, elapsed time period from sample collection to analysis, for this method is 48 hours. Chain of Custodies (COCs) and raw data are reviewed to determine if holding times were met for all samples. Positive results and nondetects will be qualified as estimated, (J) and (UJ), respectively, if holding times were exceeded. Gross holding time violations (>2X holding time) will warrant rejection, (R), of nondetects.

8.5.1.4 Quality Control

Quality control analyses (i.e., calibrations, blanks, spikes, etc.) are not specified in this method. However, if these analyses were performed by the laboratory, the following criteria will be used to evaluate the associated sample data.

Calibration

A calibration curve should be prepared by plotting absorbances of standards against known concentrations. Because the color reaction does not always obey Beer's Law, qualification of sample data based on nonlinear calibration curves may be inappropriate. Professional judgment should be used to qualify sample data when nonlinearity (calibration curve correlation coefficient <0.995) is encountered.

The percent recovery (%R) of a continuing calibration verification (CCV) standard, if analyzed, will be evaluated using an 85-115% quality control criteria. If the %R is <85%, associated sample data will be qualified as estimated, (J) and (UJ). Only positive results in the affected samples will be qualified as estimated, (J), if the CCV %R is >115%.

Blanks

If analyzed, laboratory method and field quality control blanks should be assessed for contamination. Field quality control blanks (field, rinsate, equipment, etc.) can be identified by consulting the COCs. If contamination is noted in the associated blanks, positive sample results < the maximum amount detected in the blanks will be qualified as undetected, "U." Sample digestion and moisture content factors will be taken into consideration when qualifying the associated sample data.

Spikes

A 75-125% quality control range will be used to evaluate %Rs if a spiked sample was analyzed. Associated sample results will be qualified as estimated, (J) and (UJ), when the spiked sample %R is <75%. If the %R is >125%, only positive results are impacted and qualified as estimated, (J). Associated nondetects will be qualified as rejected, (R), and positive results will be qualified as estimated, (J), if the spike %R is <30%.

Duplicates

Qualification of sample data based on duplicate precision is left to the professional judgment of the validator. Generally, an aqueous quality control limit of ±20% and a solid quality control limit of ±30% is used to evaluate the relative percent difference (RPD) between the sample and laboratory duplicate results. An aqueous quality control limit of ±30% and a solid quality control limit of ±50% is used to evaluate the RPD between field duplicate results; qualification based on field duplicate imprecision for general parameters is applied to the field duplicate pair only.

Sample Quantitation

All reported sample concentrations should fall within the range of the calibration curve. If samples having detected concentrations > the highest calibration standard were not diluted and reanalyzed, associated sample data will be qualified as estimated, (J).

The validator should verify that sample results were properly quantitated.

8.5.1.5 Deliverables Guidance

The content and format of the data package generated for this method of analysis may vary significantly depending upon the work request.

In addition to any work-request requirements (e.g., data validation memorandum), all laboratory data package quality control summary forms, laboratory summaries of sample results and laboratory method blanks, and COCs must be provided to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

8.5.2 Nitrate-Nitrite or Nitrite (EPA 300 Series Method 353.2)

8.5.2.1 Applicability

Method 353.2 is a cadmium reduction, automated colorimetric method used to determine the concentration of either nitrite or combined nitrate and nitrite in domestic and industrial effluents, and surface and saline waters. The applicable range of this method is 0.05 to 10 mg/L.

8.5.2.2 Interferences

The presence of suspended matter and high concentrations of oil and grease and some metals (i.e., iron, copper) can create interferences with this method. Samples can be filtered before analysis to minimize the problem of restricted sample flow caused by suspended matter. An organic solvent extraction and the addition of EDTA to samples can eliminate interferences from oil and grease and problematic metals, respectively.

8.5.2.3 Holding Times

Samples should be collected in plastic or glass bottles, preserved with sulfuric acid to a pH <2, and cooled to 4°C.

A 28-day holding time (elapsed time period from sample collection to analysis) is specified for analysis. Chain of Custodies (COCs) and raw data are reviewed to determine if holding times were met for all samples. Positive results and nondetects will be qualified as estimated, (J) and (UJ), respectively, if holding times were exceeded. Gross holding time violations (>2X holding time) will warrant rejection, (R), of nondetects.

8.5.2.4 Quality Control

The method does not specify the analysis of quality control measures (i.e., calibrations, blanks, spikes, etc.). However, if these analyses were performed by the laboratory, the following criteria will be used to evaluate the associated sample data.

Calibration

The calibration curve should be checked for linearity (correlation coefficient curve >0.995). In general, associated sample data are qualified as estimated, (J) and (UJ), when calibration curves are not linear. However, professional judgment should be used to qualify sample data when a nonlinear curve is encountered.

If analyzed, the percent recovery (%R) of a continuing calibration verification (CCV) standard will be evaluated using an 85-115% quality control criteria. If the %R is <85%, associated sample data will be

qualified as estimated, (J) and (UJ). Only positive results in the affected samples will be qualified as estimated, (J), if the CCV %R is >115%.

Blanks

Laboratory method and field quality control blanks, if analyzed, should be evaluated for contamination. Field quality control blanks (field, rinsate, equipment, etc.) can be identified by consulting the COCs. If contamination is noted in the associated blanks, positive sample results < the maximum amount detected in the blanks will be qualified as undetected, (U). Sample digestion and moisture content factors will be taken into consideration when qualifying the associated sample data.

Spikes

If a spiked sample was analyzed, a 75-125% quality control range will be used to evaluate %Rs. Associated sample results will be qualified as estimated, (J) and (UJ), when the spiked sample %R is <75%. If the %R is >125%, only positive results are impacted and qualified as estimated, (J). Associated nondetects will be qualified as rejected, (R), and positive results will be qualified as estimated, (J), if the spike %R is <30%.

Duplicates

Qualification of sample data based on duplicate precision is left to the professional judgment of the validator. Generally, an aqueous quality control limit of ±20% and a solid quality control limit of ±30% is used to evaluate the relative percent difference (RPD) between the sample and laboratory duplicate results. An aqueous quality control limit of ±30% and a solid quality control limit of ±50% is used to evaluate the RPD between field duplicate results; qualification based on field duplicate imprecision for general chemistry parameters is applied to the field duplicate pair only.

Sample Quantitation

All reported sample concentrations should fall within the range of the calibration curve. If samples having detected concentrations the calibration range were not diluted and reanalyzed, associated sample data will be qualified as estimated, (J).

The validator should verify that sample results were properly quantitated.

8.5.2.5 Deliverables Guidance

The content and format of the data package generated for this method of analysis may vary significantly depending upon the work request.

In addition to any work-request requirements (e.g., data validation memorandum), all laboratory data package quality control summary forms, laboratory summaries of sample results and laboratory method blanks, and COCs must be provided to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

8.6 Phosphorus (EPA 600 Series Method 365.4)

8.6.1 Applicability

Method 365.4 is a colorimetric method used to measure the concentration of total phosphorus in domestic and industrial effluents, and drinking and surface waters. The practical range of determination is 0.01 to 20 mg/L.

8.6.2 Interferences

No interferences noted in the method.

8.6.3 Holding Times

Samples are to be collected in plastic or glass containers, preserved to a pH <2 with sulfuric acid, and cooled to 4°C.

A 28-day holding time between sample collection and analysis is specified. Chain of Custodies (COCs) and raw data are reviewed to determine if holding times were met for all samples. Sample data will be qualified as estimated, (J) and (UJ), if holding times were exceeded. Gross holding time violations (>2X holding time) will warrant rejection, (R), of nondetects.

8.6.4 Quality Control

Quality control analyses (i.e., calibrations, blanks, spikes, etc.) are not specified in Method 365.4. However, if these analyses were performed by the laboratory, the following criteria will be used to evaluate the associated sample data.

8.6.4.1 Calibration

The calibration curve should be checked for linearity (correlation coefficient >0.995). In general, associated sample data are qualified as estimated, (J) and (UJ), when calibration curves are not linear.

If a continuing calibration verification (CCV) standard is analyzed, the percent recovery (%R) of the CCV will be evaluated using 85-115% quality control limits. Associated sample data will be qualified as estimated, (J) and (UJ), if the CCV %R is <85%. Only positive results in the affected samples will be qualified as estimated, (J), if the CCV %R is >115%.

8.6.4.2 Blanks

Laboratory method and field quality control blanks, if analyzed, should be evaluated for contamination. Field quality control blanks (field, rinsate, equipment, etc.) can be identified by consulting the COCs. If contamination is noted in the associated blanks, positive sample results < the maximum amount detected in the blanks will be qualified as undetected, (U). Sample digestion and moisture content factors will be taken into consideration when qualifying the associated sample data.

8.6.4.3 Spikes

If a spiked sample is analyzed, a 75-125% quality control range will be used to evaluate the spike %R. Associated sample data will be qualified as estimated, (J) and (UJ), when the spiked sample %R is <75%. If the %R is >125%, only positive results are impacted and qualified as estimated, (J). If a spike

%R is <30%, associated nondetects will be qualified as rejected. (R), and positive results will be qualified as estimated. (J).

8.6.4.4 Duplicates

Qualification of sample data based on duplicate precision is left to the professional judgment of the validator. Generally, an aqueous quality control limit of ±20% and a solid quality control limit of ±30% is used to evaluate the relative percent difference (RPD) between the sample and laboratory duplicate results. An aqueous quality control limit of ±30% and a solid quality control limit of ±50% is used to evaluate the RPD between field duplicate results; qualification based on field duplicate imprecision for general chemistry parameters is applied to the field duplicate pair only.

8.6.4.5 Sample Quantitation

All reported sample concentrations should fall within the range of the calibration curve. If samples having detected concentrations > the highest calibration standard were not diluted and reanalyzed, associated sample data will be qualified as estimated. (J).

The validator should verify that sample results were properly quantitated.

8.6.5 Deliverables Guidance

The content and format of the data package generated for phosphorus analysis may vary significantly depending upon the work request.

In addition to any work-request requirements (e.g., data validation memorandum), all laboratory data package quality control summary forms, laboratory summaries of sample results and laboratory method blanks, and COCs must be provided to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

8.7 Sulfate (EPA 600 Series Method 375.4)

8.7.1 Applicability

Method 375.4 is used to determine the concentration of sulfate in domestic and industrial effluents, and drinking and surface waters. Although all sulfate concentration ranges can be measured by this turbidimetric method, a sample aliquot should not contain more than 40 mg/L of sulfate since the suspensions lose stability at concentrations >50 mg/L. The minimum detection limit for this method is 1 mg/L.

8.7.2 Interferences

Interferences are noted from silica concentrations >500 mg/L, suspended matter, and color in samples.

8.7.3 Holding Times

Samples should be collected in plastic or glass bottles and cooled to 4°C. No preservative is necessary.

Holding time, which is specified as 28 days for this method, is defined as the elapsed time period from sample collection to analysis. Chain of Custodies (COCs) and raw data are reviewed to determine if holding times were met for all samples. Sample data will be qualified as estimated, (J) and (UJ), if holding times were exceeded. Gross holding time violations (>2X holding time) will warrant rejection, (R), of nondetects.

8.7.4 Quality Control

8.7.4.1 Calibration

The raw data will be reviewed to ensure that the following calibration requirements have been met:

- The calibration curve used for sample quantitation should consist of standards at increments of 5 mg/L in the 0 to 40 mg/L sulfate range.
- A continuing calibration verification (CCV) standard is analyzed every 3 or 4 samples.

Associated sample data will be qualified as estimated, (J) and (UJ), if the above requirements have not been met.

The calibration curve should be checked for linearity (correlation coefficient >0.995). In general, sample results associated with nonlinear calibration curves are qualified as estimated, (J) and (UJ). Professional judgment should be used to qualify sample data in instances where sample results fall outside a linear portion of the calibration curve.

The percent recovery (%R) of the CCV should be within an 85-115% quality control range. Associated sample data will be qualified as estimated, (J) and (UJ), if the CCV %R is <85%. Only positive results in the affected samples will be qualified as estimated, (J), if the CCV %R is >115%.

8.7.4.2 Blanks

Laboratory method blanks (other than the blank used for the calibration curve) should be analyzed and evaluated for contamination. The COCs should be consulted to determine if any field quality control blanks (field, rinsate, equipment, etc.) are associated with the samples. If contamination is noted in the associated blanks, positive sample results < the maximum amount detected in the blanks will be qualified as undetected, (U). Sample digestion and moisture content factors will be taken into consideration when qualifying the associated sample data.

8.7.4.3 Spikes/Duplicates

The method does not require the analysis of spikes or duplicates. However, if these quality control (QC) analyses were performed by the laboratory the following criteria will be used to evaluate the associated sample data.

QC Parameter	Control Limits
Spike %R	75 - 125%
Duplicate RPD	±20% for waters or ±30% for solids

If the spike %R is <75%, the associated sample data will be qualified as estimated, (J) and (UJ). Only positive results will be qualified as estimated, (J), when the spike %R is >125%. Associated nondetects will be qualified as rejected, (R), and positive results will be qualified as estimated, (J), in the event that the spike %R is <30%.

Generally, associated sample results are qualified as estimated, (J) and (UJ), if the relative percent difference (RPD) between the sample and laboratory duplicate results did not meet the quality control criterion. However, in some cases, qualification of sample data based on duplicate precision is left to the professional judgment of the validator. An aqueous quality control limit of ±30% and a solid quality control limit of ±50% is used to evaluate the RPD between field duplicate results; qualification based on field duplicate imprecision for general chemistry parameters is applied to the field duplicate pair only.

8.7.4.4 Sample Quantitation

All reported sample concentrations should fall within the range of the calibration curve. If samples having detected concentrations > the highest calibration standard were not diluted and reanalyzed, the associated sample data will be qualified as estimated, (J).

The validator will verify that sample results were correctly quantitated.

8.7.5 Deliverables Guidance

The content and format of the data package generated for sulfate analysis may vary significantly depending upon the work request.

In addition to any work-request requirements (e.g., data validation memorandum), all laboratory data package quality control summary forms, laboratory summaries of sample results and laboratory method blanks, and COCs must be provided to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

8.8 Sulfides (EPA SW-846 Method 9030)

8.8.1 Applicability

Method 9030 is iodometric method used to determine the concentration of total and dissolved sulfides in excess of 1 mg/L in drinking, surface and saline waters. Acid-insoluble sulfides, such as copper sulfide, can not be measured by this titrimetric method.

8.8.2 Interferences

A main source of interference for this method is the reduction of iodine by various chemicals (thiosulfate, sulfite, and organic compounds). Samples are treated at collection with zinc acetate and sodium hydroxide to minimize interferences.

In addition, sulfides are susceptible to volatilization and reaction with oxygen which can form unmeasurable states of sulfides. Aeration should be minimized during sample collection.

8.8.3 Holding Times

Samples are preserved with zinc acetate, treated with sodium hydroxide to a pH >9, and cooled to 4°C.

Holding time is defined as the elapsed time period from sample collection to analysis. The following holding times apply to sulfide analyses:

- Unpreserved samples: Immediate analysis
- Preserved samples: 7 days

Chain of Custodies (COCs) and raw data are reviewed to determine if holding times were met for all samples. In the event that holding times are exceeded, positive results and nondetects will be qualified as estimated, (J) and (UJ), respectively. Gross holding time violations (>2X holding time) will warrant rejection, (R), of nondetects.

8.8.4 Quality Control

8.8.4.1 Calibration

The raw data will be reviewed to ensure that the following calibration requirements have been met:

- The calibration curve should consist of a blank and three standards (at a minimum).
- A new calibration curve should be performed for every hour of continuous sample analysis.
- A continuing calibration verification (CCV) standard should be analyzed every 15 samples.

Associated sample data will be qualified as estimated, (J) and (UJ), if the above requirements have not been met.

The calibration curve should be checked for linearity. Generally, associated sample data is qualified as estimated, (J) and (UJ), if the calibration curve correlation coefficient is <0.995. Professional judgment should be used to qualify sample data in cases when sample results fall outside a linear portion of the calibration curve.

An 85-115% quality control range will be used to evaluate the percent recovery (%R) of a CCV. Associated sample data will be qualified as estimated, (J) and (UJ), if the CCV %R is <85%. Only positive results in the affected samples will be qualified as estimated, (J), if the CCV %R is >115%.

8.8.4.2 Blanks

At a minimum, one laboratory method blank (other than the blank used for the calibration curve) should be analyzed per sample batch (maximum of 20 samples). The COCs should be consulted to determine if any field quality control blanks (field, rinsate, equipment, etc.) are associated with the samples. If contamination is noted in the associated blanks, positive sample results < the maximum amount detected in the blanks will be qualified as undetected, (U). Sample dilution and moisture content factors will be taken into consideration when qualifying the associated sample data.

8.8.4.3 Spikes/Duplicates

A spiked sample and spiked duplicate sample should be analyzed for every 10 samples. If a spike or duplicate spike %R is <75%, associated sample data will be qualified as estimated, (J) and (UJ). If the %R is >125%, only positive results are impacted and qualified as estimated, (J). If a spike %R is <30%, associated nondetects will be qualified as rejected, (R), and positive results will be qualified as estimated, (J).

Qualification of sample data based on duplicate precision is left to the professional judgment of the validator. Generally, a ±20% aqueous quality control limit and a ±30% solid quality control limit are used to evaluate the relative percent difference (RPD) between the spiked sample and spiked duplicate sample results. An aqueous quality control limit of ±30% and a solid quality control limit of ±50% is used to evaluate the RPD between field duplicate results; qualification based on field duplicate imprecision for general chemistry parameters is applied to the field duplicate pair only.

8.8.4.4 Sample Quantitation

All reported sample concentrations should fall within the range of the calibration curve. If samples having detected concentrations > the highest calibration standard were not diluted and reanalyzed, the associated sample data will be qualified as estimated, (J).

The validator will verify that sample results were properly quantitated.

8.8.5 Deliverables Guidance

The content and format of the data package generated for sulfide analysis may vary significantly depending upon the work request.

In addition to any work-request requirements (e.g., data validation memorandum), all laboratory data package quality control summary forms, laboratory summaries of sample results and laboratory method blanks, and COCs must be provided to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

8.9 Total Suspended Solids (EPA 600 Series Method 160.2)

8.9.1 Applicability

Method 160.2 is a gravimetric method used to determined nonfilterable residue (total suspended solids) in domestic and industrial wastes, and drinking, surface and saline waters. The optimum range of total suspended solids (TSS) determined by this method is 4 to 20,000 mg/L.

8.9.2 Interferences

Requirements for appartities and analytical techniques are specified in the method to eliminate or reduce procedural interferences. Saline waters, brines, and samples high in dissolved solids must be analyzed carefully to minimize elevated sample results.

8.9.3 Holding Times

Samples should be collected in plastic or glass containers and cooled to 4°C to reduce microbiological decomposition of solids. No preservative is needed.

Holding time is defined as the elapsed time period from sample collection to analysis. Chain of Custodies (COCs) and sample data are reviewed to determine if the 7-day holding time required by this method was met for all samples. Positive results and nondetects will be qualified as estimated, (J) and (UJ), respectively, if holding times were exceeded. Gross holding time violations (>2X holding time) will warrant rejection, (R), of nondetects.

8.9.4 Quality Control

Method 160.2 does not require specific quality control analyses (i.e., blanks, duplicates, etc.). However, if these analyses were performed by the laboratory, the following criteria will be used to evaluate the associated sample data.

8.9.4.1 Verification

If a verification standard is analyzed, the percent recovery (%R) of the standard should be within a quality control range of 90-110%. Associated sample data will be qualified as estimated, (J) and (UJ), if the verification %R is <90%. Positive sample results will be qualified as estimated, (J), if the verification %R is >110%; nondetects are not impacted.

8.9.4.2 Blanks

Laboratory method and field quality control blanks, if analyzed, should be evaluated for contamination. Field quality control blanks (field, rinsate, equipment, etc.) can be identified by consulting the COCs. Positive sample results for TSS < the maximum amount detected in the blanks will be qualified as undetected, (U).

8.9.4.3 Duplicates

Qualification of sample data based on duplicate precision is left to the professional judgment of the validator. Generally, a quality control limit of ±20% is used to evaluate the relative percent difference (RPD) between the sample and duplicate results. An aqueous quality control limit of ±30% and a solid quality control limit of ±50% is used to evaluate the RPD between field duplicate results; qualification

based on field duplicate imprecision for general chemistry parameters is applied to the field duplicate pair only.

8.9.4.4 Sample Quantitation

The validator should verify that sample results were calculated accurately. The following equation is used to calculate TSS:

$$TSS\ (mg/L) = \frac{wt_{crucible+residue} - wt_{crucible}}{vol_{sample\ aliquot\ used}} \times \frac{1{,}000\ mL}{1\ L}$$

where: wt = weight (mg)
vol = volume (mL)

8.9.5 Deliverables Guidance

In addition to any work-request requirements (e.g., data validation memorandum), all laboratory data package quality control summary forms, laboratory summaries of sample results and laboratory method blanks, and COCs must be provided to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

8.10 Total Dissolved Solids (EPA 600 Series Method 160.1)

8.10.1 Applicability

Method 160.1 is a gravimetric method used to determined filterable residue (total dissolved solids) in domestic and industrial wastes, and drinking, surface and saline waters. The optimum range of total dissolved solids (TDS) determined by this method is 10 to 20,000 mg/L.

8.10.2 Interferences

Interferences during the drying stages of the analytical procedure are observed. Samples containing high concentrations of calcium, magnesium, chloride, sulfate and bicarbonate may require longer desiccation and drying times to minimize interferences. Total residue should be limited to 200 mg to prevent entrapment of water in the evaporating dish.

8.10.3 Holding Times

Samples should be collected in plastic or glass containers and cooled to 4°C to reduce microbiological decomposition of solid matter. No preservative is needed.

Holding time is defined as the elapsed time period from sample collection to analysis. A 7-day holding time is specified by the method. Chain of Custodies (COCs) and raw data are reviewed to determine if holding times were met for all samples. Positive results and nondetects will be qualified as estimated,

(J) and (UJ), respectively, if holding times were exceeded. Gross holding time violations (>2X holding time) will warrant rejection. (R), of nondetects.

8.10.4 Quality Control

Method 160.1 does not require specific quality control analyses (i.e., blanks, duplicates, etc.). However, if these analyses were performed by the laboratory, the following criteria will be used to evaluate the associated sample data.

8.10.4.1 Verification

If a verification standard is analyzed, the percent recovery (%R) of the standard should be within a quality control range of 90-110%. Associated sample data will be qualified as estimated, (J) and (UJ), if the verification %R is <90%. Positive sample results will be qualified as estimated, (J), if the verification %R is >110%; nondetects are not impacted.

8.10.4.2 Blanks

Laboratory method and field quality control blanks, if analyzed, should be evaluated for contamination. Field quality control blanks (field, rinsate, equipment, etc.) can be identified by reviewing the COCs. Positive sample results for TDS < the maximum amount detected in the blanks will be qualified as undetected, (U).

8.10.4.3 Duplicates

Qualification of sample data based on duplicate precision is left to the professional judgment of the validator. Generally, a quality control limit of ±20% is used to evaluate the relative percent difference (RPD) between the sample and duplicate results. An aqueous quality control limit of ±30% and a solid quality control limit of ±50% is used to evaluate the RPD between field duplicate results; qualification based on field duplicate imprecision for general chemistry parameters is applied to the field duplicate pair only.

8.10.4.4 Sample Quantitation

The validator should verify that sample results were calculated accurately. The following equation is used to calculate TDS:

$$TDS\ (mg/L)\ x\ \frac{wt_{dish+residue} - wt_{dish}}{vol_{sample\ aliquot\ used}}\ x\ \frac{1{,}000\ mL}{1\ L}$$

where: wt = weight (mg)
vol = volume (ml)

8.10.5 Deliverables Guidance

In addition to any work-request requirements (e.g., data validation memorandum), all laboratory data package quality control summary forms. laboratory summaries of sample results and laboratory method blanks, and COCs must be provided to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

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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.

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. The sample tracking summary is a document maintained and prepared in accordance with the requirements outlined in Standard Operating Procedure CT-02

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 5.3).

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 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 in a timely fashion.

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.

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), accuracy of the analytical results, and the presence and appropriateness of data validation qualifiers.

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 purpose of this SOP is to ensure that all necessary hardcopy information is readily available to Quality Assurance Reviewers. Therefore, the emphasis of this SOP is the establishment and maintenance of the Database Record File. The Database Record File is an additional file to the project file. The project file shall also contain all of the information contained in the Database Record 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.

5.3 Electronic Deliverables

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. Sample tracking is addressed in SOP CT-02.

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, etc. shall be photocopied by the Project Manager (or designee) and placed into a 3-ring binder. 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, etc. are accounted for and corrective actions are taken. For example, the Chain-of-Custody may list a particular sample as S42GW1 while the analytical data summary lists the sample as S42-01 and the well construction form may list S42-MW01. 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 gone through 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 A). 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.

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 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 __/__/__."

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, 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, etc.), 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, etc. 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.

5.11 Quality Assurance Records

Quality Assurance records for the Database Record File include the intermediate and final attestations 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 closeout of a given project, records from the file 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 _

ATTACHMENT A
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 NWSC, 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 NWSC, 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 D

BROWN & ROOT ENVIRONMENTAL AUDIT CHECKLIST

Brown & Root Environmental

FIELD AUDIT CHECKLIST - February 1997

QA/QC Procedures

1. Were any field observations, deficiencies, nonconformances, or complaints recorded by the site QA/QC Officer or other personnel?
   If so, summarize below.

   _____
   _____
   _____
   _____
   _____

2. Based on personnel interview, did any variances from the project planning documents occur? If so, what were they?

   _____
   _____
   _____
   _____

3. Were field modification records pertinent to the above initiated in an appropriate manner?

   _____

4. If applicable, were corrective action plans implemented (according to proper procedure)?

   _____
   _____
   _____

5. Were field QC samples obtained with the frequency specified in the QAPP, WP, or FSP?

   _____

6. For all sites, were field duplicates submitted "blind" laboratory?

   _____
   _____

7. For all sites, are sufficient replicate aliquots of samples designated to the laboratory for the matrix spike/duplicate analyses specified in the QAPP, WP, or FSP?

   _____
   _____
   _____

Boring Samples

8. Is the drilling method specified in the WP or FSP being used?

9. Are the sampling devices designated in the WP, FSP, or applicable B&RE SOP being used?

10. In accordance with B&RE policies and field SOPs, the FOL has the authority to change drilling methods if site conditions so dictate. Did any change in drilling methods from that cited in the project planning documents occur? If so, discuss.

11. If a change in drilling methods was required, did the FOL properly document the change and the rationale for the change?

12. Were any field changes initiated by the drilling subcontractor? If so, was the FOL notified and were the changes documented?

13. Per B&RE SOP GH-1.3, Sect. 5.2.1 (hollow stem auger drilling methods), was the auger plugged until the desired sampling depth was reached? (If the sample is to be taken at a relatively deep point, the auger may be advanced without a plug to within five feet of the sample depth. Beyond that point, the procedure outlined in the SOP must be observed.)

14. If water was used to prevent blowback or plugging of the hollow stem auger, has the following been recorded:

    corollary field blank sample identification _____
    amount of water introduced _____
    amount of water recovered _____
    amount of water extracted during well development _____

15. Have all abandoned borings been backfilled as specified in the WP, FSP, or applicable SOP?

    _____
    _____

16. When applicable, was the casing cleaned before sampling? (In most cases, an inch or two of cuttings may be left in the borehole with little or no problem. However, if more than a few inches for cuttings are encountered, the borehole must be recleaned prior to attempting sampling.)

    water wash (disturbed samples above and below water table) _____
    clean-out auger (undisturbed samples below water table) _____
    dry method (undisturbed samples above water table) _____

17. Were any drilling lubricants used? If so, were the procedures cited in B&RE SOP GH-1.4, Sect. 5.5 observed?

    _____
    _____
    _____

18. Were detailed boring logs maintained by the site geologist fro each borehole? (Logging is not required if explicitly stated so in the associated FSAP.)

    _____
    _____
    _____
    _____

19. Was the following information complete on the borehole logs:
    description of materials _____
    description of samples _____
    sampling method _____
    blow counts _____
    final location for drilling _____

20. Was the following information recorded in the boring logs or the field notebook?

    <u>For soil classification from core samples</u>:

Was the USCS classification indicated per Exhibit 4-2 (attached)? _____

Were the following characteristics indicated per the relevant B&RE SOP GH-1.5 sections (attached)?

color _____
soil type _____
relative density and consistency _____
weight percentage _____
moisture _____
stratification _____
texture/fabric/bedding _____

21. If classification was performed based on soil and rock drill cuttings, were the following requirements satisfied:

were cuttings obtained from 5-foot intervals? _____
were cuttings preserved in a glass sample jar or ziploc prior to classification? _____
were any changes in color or lithology recorded? _____
were any potential fracture zones noted? _____

22. Were sample aliquots from split-spoon samplers obtained representatively?

_____
_____

23. For samples acquired by thin-walled Shelby tubes, was at least an inch of soil removed from the upper and lower ends of the tube, an impervious disk inserted at both ends, a half-inch (minimum) wax seal applied, the voids at either ends filled with inert material, plastic encaps affixed and sealed with wax in accordance with B&RE SOP requirements?

_____
_____
_____
_____

24. Were Shelby tube samples handled in accordance with the following?

up direction marked with indelible ink_____
complete sample information_____
stored vertically with same orientation as in ground_____
stored out of the sun_____

Soil Sampling

25. For surface soil samples obtained by hand auger or scoop or trowel, were the following practices followed?

    area cleared of loose debris prior to sampling _____
    location marked with numbered stake or pinflag _____
    sketch approximate locations of sample points in site notebook _____

26. If test pitting is being performed, are plan and profile sketches included in the site notebook?

    _____
    _____

27. When test pitting, did the backhoe operator immediately cease digging if any of the following conditions occurred:

    encounter of any fluid or seepage; encounter of any drums, potential waste containers, obstructions, or utility lines; encounter of distinct changes of material.

    _____

28. Describe how samples were obtained (e.g., from pit via entry, from backhoe bucket, composted in buckets) and indicate if quality standards of B&RE SOP SA-1.3, 5.1.3 were met.

    _____
    _____
    _____
    _____

29. Do the site notebook entries for test pitting operations include the following information?

    name, work assignment, location of job _____
    date of digging or trenching _____
    surface elevation_____
    depth, surface area, orientation of pit _____
    associated sample numbers _____
    method of sample acquisition _____
    type and size of samples _____
    approximate water levels after stabilization (if below water table) _____
    location and depth of any seeps encountered _____
    description of soil _____
    other pertinent info. (OVA readings, weather conditions) _____
    list of photographs _____
    contractor name, backhoe operator, sampler _____
    date and type of backfill _____

Groundwater Sampling

30. Were all monitoring wells properly developed, purged and recovered prior to sampling?

31. Were the requirement of SOP SA-1.1 met for well preparation prior to sampling wells that cannot be evacuated to dryness?

32. When applicable, were well volumes calculated as described in SOP SA-1.1, 5.3?

33. If a peristaltic pump was used to obtain Volatile Organic Compound (VOC) samples, was it verified that no degassing "bubbles" developed?

34. If samples were acquired by a pump, was the pump lowered to midscreen (middle of open section of uncased wells) for sample acquisition?

35. If samples were collected using bailers, were only bailers equipped with check balls used?

36. For samples acquired by packer assembly, was the packer positioned just above the screen (or open section for uncased wells), prior to inflating?

Surface Water and Sediment Sampling

37. In accordance with SOP SA 1-2, surface water samples taken from different depths or cross-sectional locations may be composited. However, samples collected along the length of the water course or a different times shall not be composited. If composited surface water samples were obtained, was the above rule observed?

38. Per SOP SA 1-2; it is preferable to sample larger streams (and rivers) by compositing a sample from (1) just below the surface, (2) at mid-depth, (3) just above the bottom. If applicable, was this practice observed?

39. SOP SA 1-2, states that it is preferable to obtain surface water samples from a stream area that is well mixed. If applicable, was this observed?

_____
_____
_____
_____

40. For larger streams and river surface water samples, were DO, pH, temperature, and conductivity recorded for each aliquot as well as the whole composite per SOP SA-1.2?

_____
_____
_____

41. If applicable, were lakes, ponds, impoundments, and reservoirs sampled using the vertical composite approach listed in audit question No. 38 above?

_____
_____
_____
_____

42. Were DO, pH, temperature, and conductivity recorded for each aliquot as well as the whole composite?

_____
_____
_____
_____
_____

43. If applicable, did estuary sampling endeavors include the following:

samples obtained during slack tide _____
vertical salinity measurements (1-5' increments) _____
vertical dissolved oxygen profile _____
vertical temperature profile _____

44. Were specific conductance and temperature measured for each surface water obtained?

_____
_____
_____
_____

45. SOP SA-1.2, 5.3.5 states that "Even though the containers used to obtain the samples are previously laboratory cleaned, it is suggested that the sample container be rinsed at least once with the water to be sampled before the sample is taken." This is not applicable when containers are provided pre-preserved. If applicable, was this practice observed?

_____

_____

46. SOP SA-1.2, 5.3.5 states that "For sampling running water, it is suggested that the farthest downstream sample be obtained first and that subsequent samples be taken as one works upstream." Furthermore, the SOP states that work should be directed from "zones suspected of low contamination to zones of high contamination". If applicable, where these practices observed?

_____

_____

47. In accordance with SOP SA-1.2, 5.4.5, sampling at the surface should never be performed unless specifically sampling for a known constituent which is immersible and on top of the water.. Sample containers should be inverted, lowered to the approximate sample depth, then positioned at an approximate 45-degree angle with the mouth of the bottle facing upstream in order to acquire the sample. If applicable, was this technique observed?

_____

_____

Calibration and Use of Field Monitoring Equipment

48. Were the following calibration criteria observed:

calibration according to manufacturer's instructions _____
calibration only by qualified individuals _____
calibrated and operationally checked prior to project assignment _____
use of certified/traceable standards _____
calibration documented _____
if applicable, maintenance documented _____

49. For Photoionization Detectors (PIDs), is the proper ev lamp (e.g., 9.5, 10.2, 11.7) installed?

_____

_____

_____

_____

50. Because PIDs will not respond to methane or hydrogen cyanide, confirm that the instrument is not being used for this purpose, or for the detection of combustible gases or oxygen deficiency.

_____

_____

51. Confirm that proper PID Start-up and Shut-down procedures are performed as required.

_____

_____

_____

_____

52. Has PID UV light source window cleaning been conducted as required?

_____

53. Has the PID ionization chamber been cleaned as required?

_____

54. Has the PID unit been recharged after every use?

_____

_____

Equipment Decontamination Procedures

55. Has an adequate pre-determined area for steam-cleaning of equipment been established?

_____

_____

56. Is the decontamination (decon) area lined and/or bermed?

_____

_____

57. Is equipment decontaminated by steam-cleaning as required (e.g., transport vehicles, drill rigs, backhoes, downhole tools, augers, well casings, screens)?

_____

_____

58. Was steam-cleaning conducted:

prior to commencement of field activities? _____
between boring/pit locations? _____
at the end of field activities? _____

59. The sequence of solvents used is contingent upon the target analytes of concern (and Health & Safety considerations). Is the decon sequence outlined in the project planning documents being strictly observed?

_____

_____

60. Verify that all sampling equipment not subject to steamcleaning (e.g., trowels, mixing bowls, bailers, etc.) are subjected to decontamination per the sequence outlined in the project planning documents.

_____

_____

61. Have all water level indicators been contaminated via (1) potable water rinse, (2) deionized water rinse, (3) acetone/methanol (or by substitution, isopropanol for both), (4) deionized water rinse per SOP SF-2.3, 5.2.1?

_____

_____

Waste Handling Procedures

62. Were cuttings or fluids disposed of in accordance with project planning document (i.e., discharged to ground, drummed, or tanked)?

_____

63. Do the project planning documents provide for the disposal of Personal Protective Equipment (PPE) by double-bagging and discard?

_____

64. By what method are PPE disposed of?

_____

65. If applicable, were used spill-containment materials containerized or otherwise acceptably disposed of?

_____

_____

Sample Handling

66. Are the appropriate containers provided by the laboratory being used for each fractional type of sample?

67. Has a Trip Blank been submitted with each cooler of VOC samples?

68. Has the Ambient Temperature blank been handled properly and one submitted with each cooler of samples?

69. Have equipment rinsate blanks of the proper type and frequency been obtained?

70. Have Field Blanks been obtained from water sources applicable to the field effort?

71. Have the rinsate and field blanks been designated for the same analyses as the associated samples?

72. Have all samples been properly preserved in accordance with the project planning documents?

73. Is field filtration conducted in accordance with the requirements of SOP SA-6.1?

74. If applicable, have the hazardous sample packaging and shipping procedures outlined in SOP SA-6.1 been followed?

75. Has sample custody been maintained with regard to the following criteria:

A sample is under an individual's custody if:

- it is in the individual's actual possession
- it is in the individual's view after
- I t was locked up to prevent tampering
- it was placed in a designated and identified secure area

(The sample remains in the individual's custody until it is entrusted to a laboratory courier or commercial express carrier.)

Documentation

76. Are all sample logs complete (i.e., containing all information stipulated in SOP SA-6.3)?

77. Have chain-of-custody (COC) forms been filled out for all samples, including field quality control samples and samples designated for **on-site** analysis?

78. Have the COC forms been signed by the appropriate individual at each step that the samples are relinquished?

79. Have the COC forms been filled-out using black waterproof ink?

80. If the COC form was corrected, was a line drawn through the information and was the change dated and initialed? (Use of white-out or erasure is not permitted.)

81. Have the appropriate analyses (per the project planning documents) been properly designated for each sample on the chain-of-custody form?

82. Have all sample labels been filled out appropriately and completely?

83. Have all sample labels been filled out using indelible ink?

84. Have the samples been identified according to the scheme depicted in the project planning documents?

85. Do the sample identifications agree between the sample log, field notebook, sample label and chain-of-custody form?

86. When applicable, have the name of the photographer, date, time, site location, and site description been entered sequentially into the site logbook as documentative photographs of the sampling been taken?

87. Where samples have been split with a private party or government agency, have Receipt of Samples forms been filled-out and signed?

88. Has the following information (at minimum) been recorded in the site logbook:

- arrival/departure of site visitors
- arrival/departure of equipment
- sample pickup, COC form nos., carrier company, time
- sampling activities/sample logsheet nos.
- start/completion of boreholes, trenches, monitoring wells
- health and safety issues

89. Is the site logbook a bound notebook with consecutively numbered pages that cannot be easily removed?

_____

_____

90. As required by SOP SA-6.3, does the cover of the site logbook contain the following information?

project name _____
project number _____
contractor (or Teaming firm) name _____
sequential book number _____
start date _____
end date _____

91. As required by SOP SA-6.3, has the following information been recorded at the beginning of each day?

date _____
start time _____
weather conditions _____
all field personnel present _____
any visitors present _____

92. Do the site logbook entries summarize the daily activities and refer to other site notebooks or logsheets where applicable?

_____

93. Have all site logbook entries been made in black indelible ink?

_____

_____

94. If a logbook entry was corrected, was a line drawn through the information and was the change dated and initialed? (Use of white-out or erasure is not permitted.)

_____

_____

95. Did the individual making the logbook entry signed it?

_____

96. Did the Field Operations Leader sign all logbook pages utilized that day at the end of each day?

_____

_____