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REVISION 2
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Quality Assurance Project Plan
for
Base-wide Background Soil Investigation

Naval Surface Warfare Center Crane
Crane, Indiana

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

Contract Task Order 0083

April 2000

REVISION 2
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WORK PLAN
FOR
BASE-WIDE BACKGROUND SOIL INVESTIGATION

NAVAL SURFACE WARFARE CENTER, CRANE DIVISION
CRANE, INDIANA

COMPREHENSIVE LONG-TERM
ENVIRONMENTAL ACTION NAVY (CLEAN) CONTRACT

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ACRONYMS

%R	Percent Recovery
CLP	U.S. EPA Contract Laboratory Program
COC	Chain of Custody
DE	Depositional Environment
DQO	Data Quality Objective
EDQL	Ecological Data Quality Level
EMR	Environmental Monitoring Report
FOL	Field Operations Leader
FTMR	Field Task Modification Request
GFAAS	Graphite Furnace Atomic Absorption Spectroscopy
IAS	Initial Assessment Study
ICP/ES	Inductively Coupled Plasma /Emission Spectroscopy
ICP/MS	Inductively Coupled Plasma/Mass Spectrometry
IDEM	Indiana Department of Environmental Management
IDL	Instrument Detection Limit
LCS	Laboratory Control Sample
MS	Matrix Spike
NFESC	The Naval Facilities Engineering Service Center
NFG	National Functional Guidelines
NIST	National Institute for Standards and Technology
NSWC	Naval Surface Warfare Center
PARCC	Precision, Accuracy, Representativeness, Comparability, and Completeness
PDS	Post Digestion Spike
PE	Performance Evaluation
PRG	Preliminary Remediation Goal
QA	Quality Assurance
QA/QC	Quality Assurance/Quality Control
QAM	Quality Assurance Manager
QAPP	Quality Assurance Project Plan
QC	Quality Control
RBSL	Risk Based Screening Level
RCRA	Resource Conservation and Recovery Act
RFI	RCRA Facility Investigation

RL	Reporting Limit
SDG	Sample Delivery Group
SOP	Standard Operating Procedure
SSL	Soil Screening Level
SWMU	Solid Waste Management Unit
TAL	U.S. EPA Contract Laboratory Program Target Analyte List
TOM	Task Order Manager
TtNUS	Tetra Tech NUS, Inc.
U.S. EPA	United States Environmental Protection Agency
UTL	Upper Tolerance Limit
WP	Work Plan

1.0 PROJECT DESCRIPTION

This soils background investigation is being conducted in support of applicable Resource Conservation and Recovery Act (RCRA) Corrective Action requirements, including the need for RCRA facility investigations (RFIs) and other related environmental investigations to be conducted at the Naval Surface Warfare Center (NSWC) Crane, Crane, Indiana. The soils background investigation will provide data on metal concentrations in NSWC Crane soils not impacted by Site operations at Solid Waste Management Units (SWMUs). Concentrations determined for specific SWMUs may then be compared with the background concentrations to determine if a contaminant release has occurred as a result of the site operations. This quality Assurance Project Plan (QAPP) describes the collection and characterization of background data.

Based on the results of SWMU investigations, decisions are made concerning potential risks to human and ecological organisms (receptors) that could be exposed to RCRA facility contaminants. The degree of risk is determined based on the frequency, duration, and nature of the exposure to contaminants. However, detailed risk assessments can be expensive and are not necessarily warranted. To minimize costs, the risk assessment decision process is structured in a step-wise manner. Decisions concerning the acceptability or unacceptability of health risk incurred through exposure to contaminants are based on one or more evaluations of contaminant concentrations and related considerations. Each step of the evaluation process is designed to maximize efficiencies by providing information concerning the need for additional, and more costly, evaluation.

The first step in evaluating inorganic chemicals, many of which occur naturally in the environment, is often a comparison of the chemical concentrations measured in site soils to their background concentrations. Background concentrations are those concentrations that would be observed in the absence of impact from site operations. In accordance with RCRA (U.S. EPA 1989a) and risk assessment guidance (U.S. EPA 1989b), if the observed concentrations are determined not to be significantly greater than the background concentrations, an operationally-related release of those contaminants may be declared not to have occurred, and the site investigation can often be terminated at that point. If observed concentrations exceed background concentrations, additional assessment may be warranted.

Before comparisons to background concentrations can be performed, a suitable set of background data must be available. The background data must be representative of the background chemical concentrations. This means that samples collected for a background data set must be collected from areas that have not been affected by chemical releases associated with site activities. The background

samples must also have geologic, chemical, biological and physical characteristics similar to those of the SWMU samples to which they are compared (U.S. EPA, 1995).

To the extent possible, historical background data are used to maximize efficiencies because use of historic data minimizes the amount of new data that must be generated. These historical background data may have been generated during other investigations. A judgment must be made as to whether historical data, alone, are sufficient for compiling a usable background data set. When additional data are needed to complete a usable background data set, additional background samples must be collected and analyzed, and the data from the recent analyses must be combined with the historical data. Thus, the quality and statistical attributes of both historical and new data must be assessed to determine if the data sets can reasonably be combined. This means that the data quality and the actual statistical data distributions are compared to assess their similarity and suitability for combination.

This Quality Assurance Project Plan (QAPP) describes the evaluation of existing data, the collection and analysis of new background soil samples, and the associated activities required for generating background inorganic chemical data for background comparisons with soil data collected at the NSWC Crane site. It presents the organization, objectives, planned activities, and specific quality assurance/quality control (QA/QC) procedures associated with sample collection and analysis for the investigation. Specific protocols for sample collection, sample handling and storage, chain-of-custody, chemical analyses, and data evaluation and assessment are described. These protocols are specified to assure the data user that the data generated during this investigation are of the expected quality and have not been compromised.

The NSWC Crane site is located in United States (U.S.) Environmental Protection Agency (EPA), Region 5, Indiana. This document was prepared to comply with the U.S. EPA Region 5 QAPP Policy (USEPA, 1998) which includes instructions for preparing RCRA investigation QAPPs. All QA/QC procedures are structured in accordance with applicable U.S. EPA Region 5 and Indiana Department of Environmental Management (IDEM) requirements, regulations, guidance, and technical standards. The Naval Facilities Engineering Service Center (NFESC) guidance document entitled “Navy Installation Restoration Laboratory Quality Assurance Guide” (NFESC, February 1996) was also used in establishing the QA/QC requirements specified in this QAPP. The NFESC guidance specifies criteria for acceptable laboratory performance and monitoring of that performance. U.S. EPA Region 5 human health risk-based screening levels (RBSLs) and ecological data quality levels (EDQLs), as well as other human health and ecological based criteria, were used in developing this QAPP. These RBSLs and EDQLs were used primarily to identify minimum detectable analyte concentrations that are anticipated to be required for future data comparisons. Where conflicts among guidance and requirements exist, the U.S. EPA Region

5 guidance and requirements were selected to take precedence unless alternative agreement was reached with the U.S. EPA Region 5.

1.1 INTRODUCTION

This QAPP has been prepared on behalf of the United States Navy Southern Division Naval Facilities Engineering Command and NSWC Crane by Tetra Tech NUS, Inc. (TtNUS). This work was performed under Contract Number N62467-94-D-0999, Contract Task Order (CTO) Number 0083. A Work Plan (WP) (TtNUS, 1999), referred to hereafter as the Work Plan, incorporating a sampling plan and a health and safety plan, has also been prepared by TtNUS. The WP has been entirely incorporated into this QAPP through specific reference. The "Example RCRA QAPP," which is included in the U.S. EPA Region 5 QAPP Policy (U.S. EPA, April 1998), was followed closely when developing this QAPP. Also followed closely was the U.S. EPA Data Quality Objectives (DQO) process (U.S. EPA, 1994).

This QAPP is specific to the generation of background data to be used in on-going and future background comparisons with concentrations of inorganic chemicals that occur naturally in the environment and is not associated directly with decisions concerning environmental risks. However, careful consideration was given to selecting chemical analysis methods that will provide data useful for background comparisons associated with the majority of risk-based SWMU investigations at NSWC Crane.

1.1.1 Overall Project Objectives and Decision Statement

The primary objective of this investigation is to collect a sufficient number of soil samples to adequately characterize, according to depositional environment (DE), grain size, and depth, the background soil concentrations of a select number of metals at the Crane site for use in background comparisons with a discriminatory power of two standard deviations (See WP Section 4.2.3). The origin of the soil and the manner in which it was deposited largely determines the chemical and mineralogical composition of that soil. The DE refers to the parent material and/or the mechanism from which the soil was formed. NSWC Crane exhibits four primary DEs, and those DEs have been targeted for this investigation. Within each depositional environment, surface soil and subsurface soil have been targeted, because surface soil chemical composition could differ significantly from subsurface soil chemical composition. Classifying soils according to depositional environment and depth is expected to provide enough subdivision of soil types to support background comparisons. However, soil grain size (e.g., clay, silt, and sand) also may significantly impact the chemistry of soils, providing another potential discriminating factor that is not necessarily well correlated to depth. Therefore, grouping of soil grain sizes into the classes of clay, silt,

and sand will be necessary to test the expectation that background soil chemical composition is correlated most closely with depositional environment and depth.

The secondary objective of this investigation is to compute the actual minimum detectable concentration difference between SWMU and background data sets. The minimum detectable difference may be established for various conditions such as different parametric data distributions, and will be tailored to the actual data. Assumptions will be made concerning site investigation data sets to permit the computation of the minimum detectable difference.

All metal concentrations will be “total metal” concentrations (U.S. EPA 1986b). These are metals that can be dissolved from the native soil matrix by digesting the sample with a hot mineral acid in accordance with SW-836 Method 3050B, or comparable methods. Method 3050A is not actually a total digestion technique for most samples in that it will not result in complete dissolution of the soil. Rather, it is a very strong acid digestion that will dissolve most chemical elements that could become environmentally available (U.S. EPA 1986b). By design, chemical elements bound in silicate structures are not normally dissolved by this method because they usually are not mobile in the environment. Method 3050A performance data are on file at Laucks Testing Laboratories, Inc. and demonstrate that this method yields acceptable recoveries for all metals of interest for this project.

This investigation is designed for quantification of metal concentrations in background soils only. It is unknown how many samples might be collected in future investigations for comparison to background data sets. This lack of information limits the ability to project the minimum number of samples required for this investigation because data set comparisons are influenced by the number of samples in each data set. Consequently, best professional judgment was used in establishing criteria for determining whether enough data of the appropriate type and quality have been collected. Section 4.2.3 of the WP presents the detailed rationale for selecting the number of samples for this background investigation.

The decision statement for this investigation is:

> Determine if enough data points of adequate quality for each analyte in each soil type have been collected. If enough data of adequate quality have been collected, no more data will be collected; otherwise the need to collect additional data will be considered. If no additional data will be collected, the smallest detectable difference between normally distributed data sets of equal variance will be computed.

1.1.2 Project Status/Phase

Two soil depths (surface soil spanning 0 to 1 ft. depth and subsurface soil spanning 2 to 6 ft. depth) and four depositional environments (Pennsylvanian aged bedrock colluvium, Mississippian aged bedrock colluvium, loess/glacial outwash, and alluvium) representing soils throughout NSWC Crane have been identified for investigation. Further, three soil grain sizes (silt, sand, and clay) have been identified as potentially contributing to significantly differing chemical compositions among soils.

The combinations of soil grain size, depth, and depositional environment are termed “soil types” which are described in Section 5.2 of the WP. The characterization of these soil types is expected to be completed in a single phase of sampling and analysis. The soil grain sizes will be used for informational purposes to assess whether soil grain size exerts significant influence on chemical composition of Crane soils within a depth stratum. The potential need for collecting additional samples in a second phase of sampling and analysis to better define the background data distributions based on soil type is recognized. Factors that may most influence the need for additional sampling include:

- Failure to achieve the completeness goals stated in Sections 3.3.2 and 3.3.3,
- Unexpectedly large concentration variability for one or more chemicals within a given depth or depositional environment that would prevent achievement of the primary investigation objective,
- Insufficiently low detection limits caused by analytical interferences, etc.,
- Failure of any one of the sample collection, handling or analysis processes, and
- Indications that the soil depths, depositional environments and grain sizes selected for investigation inadequately represent soils that will be encountered in ongoing and/or future investigations.

In a meeting with the U.S. EPA Region 5, the Navy, based on input from TtNUS, recommended analyzing background samples for polycyclic aromatic hydrocarbons and pesticides, and possibility volatile organic compounds and polychlorinated biphenyls, to use as verification that metals analyses were indeed conducted on samples collected from background populations. The U.S. EPA Region 5 recommended, instead, that samples be analyzed for metals only. Accordingly, analyses for the U.S. EPA Contract Laboratory Program (CLP) Target Analytic List (TAL) metals, lithium, strontium, thorium, and tin (as listed in Table 1-1) will be conducted in accordance with standard U.S. EPA analytical methods designed for use in characterizing soils. No speciation of individual metal analytes will be undertaken. If speciation is

necessary for an individual project, speciation of background species may be done at that time in a project-specific background investigation.

The statistical analyses to be performed on the sample data are described in detail in Section 6.2 of the WP. Section 4.2 in the WP presents the rationale behind selecting the target number of soil samples for this project.

1.1.3 QAPP Preparation Guidelines

As previously identified, this QAPP has been prepared in accordance with the "U.S. EPA Region 5 QAPP Policy) (U.S. EPA, 1998). Additional guidance regarding generation of the QAPP was obtained in a formal "pre-QAPP" meeting, held on 2 March 1999, at U.S. EPA Region 5 Headquarters Chicago, Illinois and via teleconference on 16 April 1999. Representatives from the U.S. EPA Region 5, United States Navy Southern Division Naval Facilities Engineering Command (NFEC), NSWC Crane, and Tetra Tech NUS, Inc. attended those discussions. All issues resolved in those discussions have been addressed in this QAPP. In particular, surface soils are defined for this project as soils within 1 foot of ground surface, and analytical detection limits were selected, where practical to do so, to meet the lowest applicable risk-based levels for NSWC Crane. This is explained further in Section 1.4.3.

One unresolved issue remains:

- Selection of a sample preparation method that maintains comparability among current and future data sets and provides data useful for risk assessment. Currently under discussion is the difference between the use of soil sample particle size reduction (pulverization) and no particle size reduction.

This issue centers on a desire to limit analytical uncertainty yet generate analyte concentrations that are meaningful in risk assessment background comparisons. The proposed update to SW-846 (revision 4, January 1998) indicates that sample pulverization and blending may be necessary to achieve better precision than would otherwise be achieved when comparing duplicate analyses from a given soil sample. This is supported by Appendix Q of the U.S. EPA Region 5 QAPP guidance (U.S. EPA, 1998). The attainment of improved precision through pulverization and blending is indisputable. However, pulverization of samples is viewed to render the sample data incomparable to soils in the environment for a few reasons. Pulverization, which must typically be preceded by air-drying for days, can significantly change the soil chemical balance relative to soils in the unpulverized state (Hesse, 1971). Further, human health and ecological receptors are not exposed to pulverized soils, so the pulverized soil data are likely to misrepresent the risks that the receptors incur. Finally, pulverization is expected to increase the

concentrations of major soil matrix elements such as aluminum, iron. It is also expected to artificially increase some trace metal concentrations, especially lead and arsenic, depending on the native soil composition. The elevation of major matrix element concentrations can cause increased analytical interferences that adversely affect the quantification of trace target analytes such as arsenic. The elevation of trace metal concentrations will artificially inflate the apparent risk to human and ecological receptors. While this investigation is not a risk-related investigation, the data from this investigation will be used for background comparisons with soils that are used in risk assessments. So, while it is agreed that pulverizing soils improves analytical precision, it is also believed strongly that soil pulverization limits or invalidates the comparability and representativeness of soils for risk assessment. Comparability and representativeness were critical parameters considered during planning for data collections (Section 1.5.1 of this QAPP and Section 4 of the WP) because the intended use of data from this investigation is comparison to data from other investigations. Thus, soils will not be pulverized for this investigation. To help limit the imprecision observed with soil samples, a 1 to 1.5 gram aliquot (wet weight) of soil will be digested prior to analysis rather than a 0.5 gram aliquot which is frequently used in metals analyses.

1.2 SITE/FACILITY DESCRIPTION

A detailed description of the NSWC Crane site, its geological setting and associated features can be found in Section 2.1 of the WP.

1.2.1 Location

Sections 2.1 and 4.2.2 of the WP presents the NSWC Crane site relative to surrounding cities and towns and the specific areas selected for collection of background samples, respectively.

1.2.2 Facility/Site Size and Borders

Details concerning the NSWC Crane size and borders may be found in Section 2.2 of the WP.

1.2.3 Natural and Manmade Features

Natural and manmade features for each sample collection location are provided in Section 2.0 of the WP.

1.2.4 Topography

Section 2.2 of the WP describes the topography of NSWC Crane.

1.2.5 Local Hydrology and Hydrogeology

The drainage systems, water sources, water uses, and hydrogeology of NSWC Crane, are described in detail in Sections 2.6 and 2.8 of the WP.

1.2.6 Surrounding Land Use

Details addressing human populations and their proximity to NSWC Crane, potential health risk-related exposure pathways and related information may be found in Sections 2.3 and 2.8.2 of the WP.

1.2.7 Ecological Communities and Habitats

NSWC Crane biological characterization is presented in Section 2.4 of the WP.

1.3 FACILITY HISTORY

A summary of the general history of NSWC Crane is provided here and is supplemented in Section 2.9 of the WP.

1.3.1 General History

Summaries of the activities that might have released metals are provided in Section 2.8.2 of the WP. Detailed histories of the sample collection areas used in this investigation are found in Sections 2.8.2 and 4.2.2 of the WP.

1.3.2 Past Data Collection Activities

Based on careful review of site historical data (see Sections 2.9, 4.2.1, and 4.2.2 of the WP), including records indicating the natures and locations of actual and potential chemical releases, the areas selected for background sample collection are not expected to have received metal contaminants from site operations. This assessment of no impact is based on review of NSWC Crane operational history, topography, wind patterns, aerial photographs, and proximity of proposed sampling locations to manmade features. The final sampling locations were selected because they were accessible but far enough apart to be representative of the individual soil types and are presented in Sections 2.0 and 4.0 of the WP.

A summary of the post-IAS data collection activities for the media investigated is also provided in Section 3.0 of the WP.

1.3.3 Current Status

Several deficiencies were identified in the existing background data (section 3.0 of the WP) that range from data validation concerns to the representativeness of the samples from which the data were generated. Section 3.0 of the WP describes in detail the historical evaluation and the status of the data. Only newly-collected data will be considered for inclusion in the background data set.

1.4 PROJECT OBJECTIVES AND INTENDED DATA USAGES

This section provides a detailed explanation of the project objectives. Project target parameters and intended data uses are listed in Section 1.4.1. A review of the historical data and its impact on the data quality objectives (DQOs) is presented in Section 3.0 of the WP.

For this project, it will be necessary to gather sufficient information to:

- Address data gaps identified in previous studies that indicate a need to collect additional background data.

- Characterize the data sets with regard to concentration and spatial distributions and group the data sets according to the factors of depositional environment, depth and grain size that represent the majority of concentration variability.

- Identify any qualifications that must be placed on background data set usage.

Statistical tests, as described in Section 6.0 of the WP, will be used to evaluate and characterize the background data. For this investigation, all results greater than or equal to soil-adjusted, sample-specific IDLs will be reported. Sample-specific IDLs are IDLs determined in accordance with standard CLP protocols that have been adjusted for sample size, sample dilution and sample percent moisture. Soil-adjusted IDLs are the CLP-based IDLs adjusted to account for differences between water and soil matrices. Soil matrices typically exhibit greater detection limits than water matrices because of the relative complexity of soil matrices.

Analysis results at concentrations less than the soil-adjusted, sample-specific IDLs will be reported as the value of the soil-adjusted, sample-specific IDL with a “U” flag. The “U” flag signifies that the sample was analyzed for the analyte but that the analyte was not detected at a concentration greater than or equal to the soil-adjusted, sample-specific IDL. To account for the increased variability observed for

concentrations near IDLs, reporting limits (RLs) are generated. RLs take into account additional uncertainties in the reported result that are contributed by sample preparation, analytical interferences associated with soil matrices, and long-term method variability. The RLs are based in part on best professional judgment and represent the lowest analyte concentrations that can be reported with confidence that the reported value approximates the true value. Soil-adjusted IDLs and RLs for this project are listed in Table 1-1.

The decision rule to address the primary investigation objective and to guide the sampling plan design is:

> If at least five data points of adequate quality for each analyte for each of the soil types have been collected and verified to represent soils at NSWC Crane, no more data will be collected; otherwise the need to collect additional data will be considered. Considerations to be addressed when deciding to collect additional data include the discriminatory power achievable with the collected data and the cost of collecting additional background samples *versus* collecting additional SWMU investigation samples. If no additional data will be collected, the smallest difference that can be detected with the existing background data set will be estimated, assuming normally distributed data and equal data set variances.

Data quality will be adequate if the completeness criteria of Sections 3.3.2 and 3.3.3 are satisfied and if no anomalies are identified that might compromise the usability of the data for background comparisons. Based on data quality, data use qualifications may be established upon inspection and analysis of the data. Non-detected analytes (i.e., measured concentration less than soil-adjusted, sample-specific IDL) of acceptable quality will be considered usable.

To achieve the investigation objectives, a confirmational level of analytical quality is needed. This provides the highest level of data quality necessary to address potential human and ecological risks, thus helping to assure that background data quality does not impose limitations on future environmental investigations. These analyses require full documentation of the chosen U.S. EPA SW-846 analytical methods and sample preparation steps, data packages, and data validation sufficient to provide defensible data. QC must be sufficient to define the overall precision and accuracy of these procedures.

1.4.1 Project Target Parameters

The list of target parameters is presented in Table 1-1. Other inorganic chemicals and naturally occurring organic chemicals or derivatives thereof were considered for inclusion in this investigation but were ultimately considered to extend beyond this investigation scope.

Also of critical importance to this investigation are the classifications used to determine grain size. These classifications are important because data analyses will consider grain size as a potential source of variability when assessing whether soils have been adequately characterized. The grain size classifications are defined and described in Sections 5.2.2, 5.2.3 and 5.2.4 of the WP.

Further details regarding the specific sampling and analytical programs for the background investigation are provided in Section 5.0 of the WP.

1.4.2 Field Parameters

Grain size will be determined on site by field geologists who, by virtue of their training, have competency in this area. No chemical field measurements will be made for this investigation.

1.4.3 Laboratory Parameters

Table 1-1 provides a list of the laboratory parameters and the lowest risk-based target concentrations to which future data may be compared. The listed parameters are the CLP TAL metals plus lithium, strontium, thorium, and tin. As indicated in Table 1-1, tin is included in the Appendix IX list of constituents but not in the TAL metals list. Lithium, strontium and thorium are absent from both lists but these metals are known to have been used and potentially disposed at NSWC Crane. Consideration was given to the analytical detection limits relative to regulatory limits that might be required for future investigations. The lowest regulatory limits that might apply to foreseeable investigations have been selected as points of reference for determining whether detection limits will be low enough to support future studies. The risk-based target levels presented in Table 1-1 are based on human health and ecological risk-based criteria. A tabular presentation of the human health and ecological risk-based criteria used as a basis for determining the target levels is provided in Appendix A, Table A-1. The following is a list of those risk-based target criteria:

- U.S. EPA Soil Screening Levels (SSLs) for Ingestion, Inhalation and Migration to Ground Water
- U.S. EPA Region 9 Preliminary Remediation Goals (PRGs) for Residential and Industrial Land Use

- IDEM Tier I Surface and Subsurface Soil Default Cleanup Levels for Residential and Non-residential Land Use

- U.S. EPA Region 5 Ecological Data Quality Levels for Soil

Based on the limitations of best available technology, RLs for eleven metals do not meet the associated risk-based target levels; the laboratory RLs of these analytes are marked with double asterisks (**) on Table 1-1. Of those eleven analytes, six have soil-adjusted IDLs that exceed the risk-based target level (*), although two of these six are within 7 percent of the applicable risk-based target level. These analytes were discussed during the Pre-QAPP meeting held at U.S. EPA Region 5 Headquarters on March 2, 1999. The following text summarize the rationale for the conclusions reached during the meeting with U.S. EPA Region 5, and the rationale for using the analytical methods established in this QAPP and presented on Table 1-1.

TtNUS worked closely with the subcontracted analytical laboratory to select and optimize analytical methods in an effort to attain, to the greatest extent possible, laboratory RLs at concentrations less than or equal to the risk-based target levels. Steps taken to achieve lower RLs include the use of SW-846 Method 6020 (Inductively Coupled Plasma/Mass Spectrometry [ICP/MS]) in place of SW 846 Method 6010B (ICP/Emission Spectroscopy [ICP/ES]) for some metals. Consideration was given to using other analytical methods to accommodate those cases for which future site investigations which may make use of other analytical methods. However, it was determined that the precision and accuracy data generated as part of this investigation, and generally available from the analytical methods, will be sufficient to permit performance comparisons among ICP/ES, ICP/MS and other analytical methods. Consideration also was given to reporting metal concentrations less than RLs with the understanding that U.S. EPA Region 5 is most interested in knowing that an analyte is detected and is not as interested in obtaining a precise estimate of the analyte concentration. This is consistent with the fact that precision of laboratory operations frequently is typically a minor component of overall investigation uncertainty. Further, the primary distinguishing factor in overall analytical precision, accuracy, comparability, and representativeness was judged to be not the determinative analytical method, but the sample preparation method. Graphite furnace atomic absorption spectroscopy (GFAAS) is the most likely candidate analysis method next to ICP/MS to be used to achieve the indicated detection limits. The ICP/MS detection limits are generally slightly less than those achievable with GFAAS. Because ICP/MS and GFAAS employ similar sample preparation methods, the ICP/MS data should be comparable to potential future GFAAS data.

The analysis methods that will be used in future environmental investigations are unknown. Therefore, analysis method selection for this investigation was based on the following assumptions. Method 3050A is assumed to best represent the sample preparation methods that will be used in the future because it is a standard method for soil digestion that has been in use for several years with much success. Its use will provide data that also represent metal concentrations that could be available to most human and ecological receptors under normal circumstances, although the concentrations are probably overestimates for exposure pathways other than direct ingestion. SW-846 methods or comparable methods are likely to be used for future analyses of soils at NWSC Crane because it is a RCRA site and the EPA SW-846 methods are the most commonly used environmental soil analysis methods at such sites. Most environmental laboratories are equipped to implement SW-846 soil analyses methods under routine operations and turnaround times are reasonable.

1.4.4 Data Analysis and Interpretation

Laboratory data will be used to estimate chemical concentrations within each soil type. The data will first be compiled into an electronic database by electronic transfer of the data from magnetic disks received from the laboratory. The data will be validated as described in Section 9.2.2. Additional treatment of project data is described in Section 12.0 of this QAPP and in Section 6.0 of the WP.

1.5 SAMPLING LOCATIONS

Figures 5-1, 5-2 and 5-3 of the WP depict sampling locations. Depending on the site conditions at the time of sampling, sampling locations may be changed in accordance with Section 5.2.7 of the WP and 13.1 of this QAPP

1.5.1 Rationale for Selected Sampling Locations

The rationale for selecting sampling locations is explained in detail in Section 4.2 of the WP. The number of samples selected for each soil type is based on computations used to model background data set comparisons in a simple manner. Those computations are presented in Section 4.2.3 of the WP.

Future soil sampling methods are unknown but this factor has been considered. Many standard sampling protocols exist that can be used for collecting soil samples for metals analyses. Therefore, sampling methods were selected for use based on the medium to be sampled, accessibility to the sampling locations, cost of sampling, and time required to collect samples. The sampling methods are judged to represent soil sampling methods likely to be used in future environmental investigations at NSWC Crane. The greatest concerns associated with soil sampling quality for metals analyses are representativeness of

the samples and cross-contamination through direct or indirect contact. Representativeness has been partially addressed by specifying criteria by which each soil type of interest will be classified (see Section 4.2 of the WP). Representativeness also will be addressed by carefully selecting the material to be sampled and by controlling cross-contamination. This will be controlled in the field by the sampler who will strive to collect a single soil type in each sample bottle. Cross-contamination will be controlled through the use of disposable sampling equipment and by careful adherence to sampling equipment decontamination protocols when reusable sampling equipment is used. Further, the potential for cross-contamination is not expected to be significant because samples are being collected from areas that should exhibit only background concentrations.

1.6 PROJECT SCHEDULE

This section refers to the schedule, included in Section 7.0 of the WP, for field investigation activities at NSWC Crane.

1.6.1 Anticipated Date of Project Mobilization

The earliest date on which samples are planned to be collected is 7 September 1999. Section 7.0 of the WP addresses this topic and presents the overall project schedule.

1.6.2 Task Bar Chart and Associated Timeframes

The dates of projected milestones are presented in Section 7.0 of the WP.

TABLE 1-1

SOIL ADJUSTED IDLs AND RLs
NAVAL SURFACE WARFARE CENTER
CRANE, INDIANA

Chemical[1]	Appendix IX Metals	CLP TAL Metals	Water IDL (ug/L) [2]	Water RL (ug/L) [2]	Soil Adjusted (IDL)[3] (mg/kg)	Soil RL[3] (mg/kg)	Soil Risk Based Target Level[4] (mg/kg)
Aluminum (SW-846 Method 6010B)		X	50	200	6.8	8	75,000
Antimony	X	X	1.0	1.0	0.15*	0.5**	0.1423
Arsenic	X	X	1.0	1.0	0.15	0.5**	0.38
Barium	X	X	0.5	1.0	0.15	0.5	1.04
Beryllium	X	X	0.5	1.0	0.15*	0.5**	0.1
Cadmium	X	X	0.5	1.0	0.15	0.5**	0.18095
Calcium (SW-846 Method 6010B)		X	112	1000	2.3	500	--[5]
Chromium (total)	X	X	0.5	5.0	0.15	2.5**	0.4
Cobalt	X	X	0.5	3.0	0.15*	1.5**	0.14033
Copper	X	X	0.5	2.0	0.15	1**	0.3132
Iron (SW-846 Method 6010B)		X	16	100	0.69	10	22,000
Lead	X	X	0.5	1.0	0.15	0.5**	0.45053
Lithium			0.18	10	0.09	5	1,500
Magnesium (SW-846 Method 6010B)		X	33	1000	3.3	500	--
Manganese (SW-846 Method 6010B)		X	1.5	15	0.15	1.5	3,100
Mercury (*SW-846 Method 7471A)	X	X	0.06	0.2	0.07*	0.1**[6]	0.0078
Nickel	X	X	0.5	10	0.15	5	7
Potassium (SW-846 Method 6010B)		X	147	1000	12	500	--
Selenium	X	X	1.0	1.0	0.15*	0.5**	0.02765
Silver	X	X	0.5	3.0	0.05	1.5	2
Sodium (SW-846 Method 6010B)		X	28	1000	2.6	500	--
Strontium			0.15	10	0.08	5	45,000
Thallium	X	X	0.5	1.0	0.15*	0.5**	0.04
Thorium			0.17	10	0.09	5	--
Tin	X		0.1	10	0.10	5	7.62
Vanadium	X	X	0.5	2.0	0.15	1	1.59
Zinc	X	X	5.0	10	0.15	5	6.62

* Single asterisk indicates those chemicals for which the laboratory soil-adjusted IDL exceeds the risk-based target level for the project.
** Double asterisks indicate those chemicals for which the laboratory RL exceeds the risk-based target level for the project.
1 Analyzed using SW-846 Method 6020 unless otherwise noted.
2 Maximum expected limit; for aqueous QC samples only. Actual limits may vary but shall not exceed these limits.
3 Maximum instrument detection limits *IDLs) and reporting limits (RLs) as provided by Laucks Testing Laboratories, Inc. Actual values may be less than these. Assumes 1 g of soil in 100 mL of digestate diluted five-fold to limit total dissolved solids content.
4 Value is based on human health or ecological risk-based criteria as presented in Appendix A, Table A-1.
5 Risk-based target level is not provided, since human and ecological risk-based criteria are not available for this chemical.
6 Laucks Testing Laboratories is confident that it can reliably report to this RL, even though this value is less than two times the IDL.
TAL = Target Analyte List

2.0 PROJECT ORGANIZATION AND RESPONSIBILITY

The project organization for the NSWC Crane background soil investigation is provided in Section 1.1 of the WP. A project organization chart is provided, and management, quality assurance, laboratory, and field responsibilities are presented.

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 that are technically sound and will adequately support future background comparisons. Intended data uses are described in Section 1.4 of this QAPP. Specific procedures for sampling, chain-of-custody, laboratory instrument calibration, laboratory analysis, reporting of data, internal quality control (QC), audits, preventive maintenance of field and laboratory equipment, and corrective action are described in other sections of this QAPP.

The PARCC parameters (precision, accuracy, representativeness, comparability, and completeness) are qualitative and quantitative statements regarding the quality characteristics of the data used to support project objectives and ultimately, environmental decisions. These parameters are presented 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 of this QAPP.

3.1 PRECISION

3.1.1 Definition

Precision is a measure of the degree to which two or more measurements are in agreement and describes the reproducibility of measurements of the same parameter for samples analyzed under similar conditions. A fundamental tenet of using precision measurements for quality control is that precision will be bounded by known limits. Results outside the limits trigger corrective actions.

Because of the inherent and unknown heterogeneity of soil samples, the precision of field duplicate samples will not be used for quality control, but it will be compared to laboratory precision estimates for information to gain a perspective on the natural heterogeneity of the soil. The equation for estimating precision for duplicate samples is provided in Section 12.2. In addition, Section 6.2 of the WP addresses statistical analyses of project data for evaluation as background data. As part of those evaluations, statistical parameters such as data set variances will be computed that provide direct insight into the variability of metals data in background soils.

3.1.2 Field Precision Objectives

Field precision is assessed through the collection and measurement of field duplicates at a rate of 1 duplicate per 10 environmental samples. This precision estimate encompasses the combined uncertainty associated with sample collection, homogenization, splitting, handling, storage, digestion and analysis. In contrast, precision estimates obtained from analysis of duplicate laboratory samples incorporate only homogenization, subsampling, digestion and analysis uncertainties. Consequently, the field precision estimates (i.e., RPD values) should equal or exceed the laboratory precision estimates on average for each analyte. If field duplicate precision is significantly different from laboratory duplicate precision, the underlying cause will be investigated. Considerations given to this effort include:

- The scale of subsampling for laboratory precision estimates relative to the scale of field duplicate sample size.
- Analytical measurement precision.
- Precision for repeat analysis of the same solid laboratory control sample.
- Sample grain size relative to LCS grain size.
- Natural soil heterogeneity

No criteria for acceptability have been developed for the comparison of analyte concentration precision estimates because of the unknown soil heterogeneity. However, review of background data from other NSWC Crane investigations, suggest that most RPD values (see Section 12.2) will be less than 50%.

3.1.3 Laboratory Precision Objectives

Laboratory precision QC samples (i.e., laboratory duplicates) will be analyzed with a minimum frequency of 5 percent (i.e., 1 QC sample per 20 environmental samples). Laboratory precision for inorganic analyses is measured via comparison of relative percent difference (RPD) values with precision control limits specified in Table 3-1.

3.2 ACCURACY

3.2.1 Definition

Accuracy is the degree of agreement between an observed value and an accepted reference value. This parameter is assessed for the analysis process by measuring spiked samples or well-characterized samples of certified analyte concentrations (e.g., LCSs). The intent of accuracy measurements is to provide indications of biases in the sample handling and analysis processes. The equation for

determining accuracy of an individual matrix spike for this project is provided in Section 12.1. Also used as an indication of biases is measurement of blank samples that ideally should exhibit no detectable analytes**.** The equation in Section 12.1 does not apply to blank samples, however, because division by zero (the expected amount or added amount) causes the calculated value to be infinite. Instead, acceptance criteria are designed to limit the tolerable amount of contamination while recognizing that non-zero results for blanks are likely, if only because of random error in the measurement process. The bias computations for individual matrix spikes will be used to control the analysis process by triggering corrective actions as specified in Section 8.2.

3.2.2 Field Accuracy Objectives

Accuracy requirements for field measurements are typically ensured through control over the sample collection and handling, and routine instrument calibration. However, no chemical field measurements are intended for this investigation. Accuracy is also typically monitored through the use of blanks to detect cross-contamination, and by monitoring adherence to procedures that prevent sample contamination or degradation. Equipment rinsate blanks shall be collected for this investigation to assess cross-contamination via sample collection equipment, although the potential for cross-contamination from sampling background soil is assumed to be insignificant. Ambient condition blanks will not be collected unless site conditions during sampling indicate a need to assess infiltration of airborne contaminants into sampling containers. Source water blanks will be collected to monitor the purity of water used to decontaminate sampling equipment. Accuracy also shall be assured qualitatively through adherence to all sample handling, preservation, and holding time requirements.

3.2.3 Laboratory Accuracy Objectives

Accuracy in the laboratory is measured through the comparison of a spiked sample result to a known or calculated value and is expressed as a percent recovery (%R). Percent recoveries are derived from the analysis of known amounts of compounds spiked into actual samples (i.e., matrix spike analysis and post-digestion spikes) or of LCS analyses. Matrix spike analyses measure the combined accuracy effects of sample matrix, sample preparation and sample measurement. Post-digestion spikes (PDSs) measure the accuracy of the analytical measurement. LCSs measure the accuracy of laboratory operations in the absence of sample matrix effects. LCS and MS analyses are performed at a frequency no less than 1 per 20 associated samples of like matrix. Laboratory accuracy is assessed via comparison of calculated %R values to accuracy control limits specified in Tables 3-1 and 3-2.

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 often expressed as a percentage, but for this project the completeness requirement is a specification of the fewest usable results generated during the investigation.

Validation will be performed for 100 percent of the laboratory data for this investigation based on the requirements of the analytical methods and this QAPP. To the extent practicable for SW-846 analyses, validation also will be performed in accordance with the U.S. EPA Region 5 Standard Operating Procedures (SOPs) for Validation of CLP (Contract Laboratory Program) Inorganic Data (U.S. EPA Region 5, 1993b) and the U.S. EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review (U.S. EPA, 1994b). Data rejected as a result of these validation processes will be scrutinized further to determine if the severe deficiencies are verifiable as unreliable, unusable data. No data judged to be unusable will be included in the background data set analyses.

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. No field chemical measurements will be made during this investigation. However, soil grain size will be characterized for each sample collected. The means of monitoring completeness is specified in section 12.3 of this QAPP.

For this project, within a DE, subsurface soil samples will be collected according to grain size. Surface samples will not be collected according to grain size. See Section 4.2.1 of the WP for a complete discussion of the sampling plan rationale. The following numbers of samples from each depositional environment shall be required unless the desired number of samples of a given soil type are unavailable for collection:

- Subsurface clay: 5 samples
- Subsurface silt: 5 samples
- Subsurface sand: 5 samples
- Surface soil: 5 samples

Anticipated conditions that may lead to unavailability of a particular soil type are:

- Shallow bedrock (i.e., less than 6 ft. depth)
- Encountering a saturated zone at a depth shallower than 6 ft.
- Inability to advance the auger to the desired depth because of an obstruction.
- No soil grain size of the desired type

Failure to collect a particular soil type because of unavailability will be substantiated by field records.

3.3.3 Laboratory Completeness Objectives

Laboratory completeness is a measure of the amount of usable, valid laboratory measurements per matrix obtained for each of the 24 target metals. Usable, valid results are those that are judged, after data assessment, to represent the background soil metal populations and which have not been disqualified for use through data validation and data assessment.

Laboratory completeness objectives are the same as field completeness objectives (See first set of bullets in Section 3.3.2).

With four depositional environments, the total number of non-QC samples collected is expected to be no less than 40 (only one grain size encountered in each DE) and no greater than 80 (all grain sizes encountered in each DE).

Failure to achieve this completeness goal will not necessarily invalidate the results of the investigation although the statistical power achievable in future data comparisons may be reduced. Because statistical power depends on the number of samples in both the background data set and the site investigation data set, the collection of proportionately more samples may be required in future site investigations to achieve the required statistical power. Thus, completeness deficiencies in the background data set may be compensated by collecting additional samples in SWMU investigations. Qualifications on the use of the background data set caused by completeness considerations will be documented in the final report.

3.4 REPRESENTATIVENESS

3.4.1 Definition

Representativeness is an expression of the degree to which the data accurately and precisely represent a characteristic of a population or environmental condition existing within a soil type. Use of standardized

sampling, handling, preparation, analysis, and reporting procedures ensures that the final data accurately represent actual site conditions. An assessment of representativeness will be made during data review to determine whether each datum belongs to the observed data distribution through outlier testing. The statistical tests to be used are described in detail in Section 6.2 of the WP. Any anomalies will be investigated to assess their impact on statistical computations and future data set use.

3.4.2 Measures to Ensure Representativeness of Field Data

Representativeness depends upon the proper design of the sampling program and will be satisfied by ensuring that the WP is followed and that proper sampling techniques are used. Specifically, strict adherence to soil type descriptions and care to ensure that samples representing a single soil type is placed into each sample container will be ensured during sample collection.

3.4.3 Measures to Ensure Representativeness of Lab Data

Representativeness in the laboratory is ensured or evaluated by using the proper analytical procedures, meeting sample holding times, and analyzing and evaluating field duplicate samples relative to laboratory duplicates. The sampling plan for the background investigation is designed to provide data representative of NSWC Crane soils not affected by site operations. During development of this plan, consideration was given to past waste disposal practices, aerial photographs, existing analytical data, physical setting and processes, depositional environments, spatial coverage of the proposed sampling locations, accessibility to sampling locations, and constraints inherent to the RCRA program. The rationale of the sampling network is presented in detail in Section 4.2 of the WP.

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 by ensuring that reporting limits and detection limits are sufficiently low to satisfy project detection and quantitation criteria. The reporting limits anticipated for this project are presented in Table 1-1. Additionally, consideration is given to seasonal conditions and other environmental variations that could exist to influence analytical results. It is emphasized that use of comparable sampling and analysis methods in future investigations should limit the need to consider biases when making data set comparisons.

3.5.2 Measures to Ensure Comparability of Field Data

Comparability depends upon the proper design of the sampling program and will be satisfied by ensuring that the WP is followed and that proper sampling techniques are used. The rationale behind the sampling plan design is found in Section 4.2 of the WP.

3.5.3 Measures to Ensure Comparability of Laboratory 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. The units used for the laboratory measurements are further explained in Section 9.1.2 of this QAPP.

3.6 DECISION RULE

3.6.1 Definition

A Decision Rule is a statement which allows for a course of action or non-action to be taken, based on assumptions made to draw out and test its logical or empirical consequences.

3.6.2 Decision Rule Objectives

Decision levels, expressed as human health or ecological target decision levels, for each critical measurement parameter are specified in Table 1-1 of this QAPP. The decision rule for this investigation is presented in Section 1.4.

3.7 LEVEL OF QUALITY CONTROL EFFORT

Field duplicate, rinsate blank, source water blank, calibration blank, method blank, laboratory duplicate, laboratory control, and MS samples will be analyzed to assess the quality of the data resulting from the field sampling and analytical programs. Temperature blanks will be included in each cooler submitted to the laboratory. Laboratory QC samples are addressed in Sections 6.0 and 8.0 of this QAPP. Section 6.0 addresses instrument calibrations; Section 8.0 addresses non-calibration analytical QC. Field QC samples consist of field duplicates, equipment rinsate blanks, source water blanks and, at the discretion of the FOL, ambient condition blanks. Laboratory QC includes a host of other quality checks performed during sample preparation and analysis. Information gained from these analyses further characterizes the level of data quality obtained to support project goals. Each type of field QC sample undergo the same preservation, analysis, and reporting procedures as the related environmental samples. The types

of field and analytical QC to be used for this project are described below. Table 5-5 of the WP indicates the anticipated total number of field QC samples to be collected.

Source water blanks are obtained by sampling the analyte-free water and/or potable water source(s) used for decontaminating sampling equipment. Source water blanks are used to determine whether the analyte-free water (used for sampling equipment decontamination procedures) or the potable water (used for steam cleaning) may be contributing to sample contamination.

Equipment rinsate blanks or **rinsate blanks** are obtained under representative field conditions by collecting the rinse water generated by running analyte-free water through sample collection equipment after sampling and decontamination and prior to use. These blanks will be collected to indicate the potential for sample cross-contamination through the use of improperly cleaned sampling equipment.

Ambient condition blanks are samples of deionized water poured from one container to another in the field to detect the infiltration of airborne contaminants into field samples. These samples will be used at the discretion of the FOL if the FOL believes that such infiltration is a possibility.

Field duplicates will be collected and analyzed for chemical constituents to measure the cumulative uncertainty (i.e., precision) of the sample collection, splitting, handling, storage, preparation and analysis operations, as well as natural sample heterogeneity that is not eliminated through simple mixing in the field. Field duplicates are two samples prepared by mixing a mass of soil sample and splitting it into two separate sample containers that are labeled as individual field samples.

Laboratory control samples serve to monitor the overall performance of each step during the analysis, including the sample preparation. These are solid samples that contain concentrations of analytes that are known with a specified degree of certainty.

Laboratory duplicates will be analyzed for chemical constituents to measure the cumulative uncertainty (i.e., precision) of the sample handling, subsampling, preparation and analysis operations within the laboratory, as well as sample heterogeneity that is not eliminated through simple mixing in the laboratory. Laboratory duplicates are two subsamples obtained by the laboratory analyst after mixing the sample.

Laboratory method blanks or preparation 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. Native soils devoid of acid leachable metals do not exist. Therefore, a method blank for inorganic soil sample analysis consists of an aliquot of

analyte-free water that is subjected to the same preparation and analysis procedures as the environmental samples undergoing analysis. The aqueous results are normalized to a fictitious soil sample and presented on a dry weight basis assuming 100% solids.

Matrix spikes (MSs) are environmental samples to which known quantities of analytes are added prior to sample digestion. These samples provide information about the heterogeneity of the samples as well as the effect of the sample matrix on the sample digestion and measurement methodology. Matrix spiking concentrations are identified in SOPs LTL-7105 or LTL-7202 (TAL metals except mercury, plus lithium, strontium, thorium, and tin), and LTL-7501 (Hg only).

Post digestion spikes (PDSs) are similar to MSs except that the sample digestate, rather than the original soil sample, is spiked. Comparing %R for PDSs and MSs helps to identify where in the analytical process accuracy problems are occurring.

Temperature Blanks These blanks are vials of water inserted into each cooler. The temperature of the temperature blank is measured prior to shipping the cooler to the laboratory and upon receipt at the laboratory to verify that samples were properly cooled during transit.

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TABLE 3-1

QUALITY CONTROL LIMITS
LABORATORY MATRIX/POST DIGESTION SPIKE AND DUPLICATE SAMPLES
NAVAL SURFACE WARFARE CENTER
CRANE, INDIANA

Chemical (Analysis Method[1])	Solid Matrix	
	Accuracy (%R)[2]	Precision (RPD)[3]
Aluminum (SW-846 Method 6010B)	75-125	50%
Antimony	75-125	50%
Arsenic	75-125	50%
Barium	75-125	50%
Beryllium	75-125	50%
Cadmium	75-125	50%
Calcium (SW-846 Method 6010B)	75-125	50%
Chromium (total)	75-125	50%
Cobalt	75-125	50%
Copper	75-125	50%
Iron (SW-846 Method 6010B)	75-125	50%
Lead	75-125	50%
Lithium	75-125	50%
Magnesium (SW-846 Method 6010B)	75-125	50%
Manganese (SW-846 Method 6010B)	75-125	50%
Mercury (SW-846 Method 7471A)	75-125	50%
Nickel	75-125	50%
Potassium (SW-846 Method 6010B)	75-125	50%
Selenium	75-125	50%
Silver	75-125	50%
Sodium (SW-846 Method 6010B)	75-125	50%
Strontium	75-125	50%
Thallium	75-125	50%
Thorium	75-125	50%
Tin	75-125	50%
Vanadium	75-125	50%
Zinc	75-125	50%

1 Unless otherwise specified, samples will be analyzed by SW-846 method 6020.
%R = Percent Recovery
RPD = Relative Percent Difference

2 These acceptance limits apply to spikes that augment the native sample analyte concentration by at least 25%.

3 These acceptance limits apply to original and duplicate sample concentrations >5X RL. If one of the results is <5XRL, the acceptance criterion is ±4XRL. If one or the other results is a non-detect, the reported %RPD will be 200%.

TABLE 3-2

**QUALITY CONTROL LIMITS
LABORATORY CONTROL SAMPLES
NAVAL SURFACE WARFARE CENTER
CRANE, INDIANA**

Chemical[1]	Solid Matrix Accuracy (%R)
Aluminum (SW-846 Method 6010B)	Specified by LCS supplier
Antimony	Specified by LCS supplier
Arsenic	Specified by LCS supplier
Barium	Specified by LCS supplier
Beryllium	Specified by LCS supplier
Cadmium	Specified by LCS supplier
Calcium (SW-846 Method 6010B)	Specified by LCS supplier
Chromium (total)	Specified by LCS supplier
Cobalt	Specified by LCS supplier
Copper	Specified by LCS supplier
Iron (SW-846 Method 6010B)	Specified by LCS supplier
Lead	Specified by LCS supplier
Lithium	Specified by LCS supplier
Magnesium (SW-846 Method 6010B)	Specified by LCS supplier
Manganese (SW-846 Method 6010B)	Specified by LCS supplier
Mercury (SW-846 Method 7471A)	Specified by LCS supplier
Nickel	Specified by LCS supplier
Potassium (SW-846 Method 6010B)	Specified by LCS supplier
Selenium	Specified by LCS supplier
Silver	Specified by LCS supplier
Sodium (SW-846 Method 6010B)	Specified by LCS supplier
Strontium	Specified by LCS supplier
Thallium	Specified by LCS supplier
Thorium	Specified by LCS supplier
Tin	Specified by LCS supplier
Vanadium	Specified by LCS supplier
Zinc	Specified by LCS supplier

1 Unless otherwise specified, samples will be analyzed by SW-846 Method 6020.

4.0 SAMPLING PROCEDURES

All field sampling locations, QC samples and sample collection procedures have been selected to facilitate achievement of project objectives. Site maps showing sampling locations and the rationales for selecting the locations are provided in Sections 4.0 and 5.0 of the WP. The sampling procedures, which provide stepwise instructions for collecting each soil sample, as well as SOPs governing related field activities are detailed in Section 5.2 of the WP. Copies of the SOPs are included as Appendix C to the WP. Collection of QC samples is addressed in Section 5.3 of the WP. Sample containers shall be obtained from the laboratory as certified clean. Table 5-4 of the WP presents sample bottle, preservation and holding time requirements.

5.0 CUSTODY PROCEDURES

Documented sample custody is one of several factors that 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. While samples collected during this investigation will not be used directly in making decisions concerning risk of exposure to contaminants, they will be used to identify the existence of metal analytes greater than background concentrations in on-going and future environmental investigations. Consequently, these data will be subject to the same custody procedures as any other SWMU investigation data.

Sample custody is addressed in three parts: field sample collection, laboratory analysis, and final evidence files. Final evidence files, including all original laboratory reports and purge files, are maintained under document control in a secure area. A sample or evidence file is under custody when any one of the following conditions is satisfied:

- The item is in the actual physical possession of an authorized person.
- The item is in view of the person after being in his or her possession.
- The item was placed in a secure area to prevent tampering.
- 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, sample 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. Laboratory custody procedures will ensure that sample integrity is not compromised from the time of receipt at the laboratory until final data are reported to Tetra Tech NUS. This requires that the laboratory control all sample handling and storage conditions and circumstances. Custody procedures apply to all environmental and associated field QC samples obtained as part of the data collection system.

5.1 FIELD CUSTODY PROCEDURES

The FOL (or designee) is responsible for the care and custody of the samples collected until they are relinquished to the laboratory or entrusted to a commercial courier. COC forms are completed for each

sample shipment. The forms are filled out legibly with waterproof ink, and are signed (and dated) by the sampler. Pertinent notes are also indicated on the COC form. Information similar to that contained in the COC form is 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 returned by the analytical laboratory for inclusion in the final evidence file.

Site conditions during sampling and the care with which samples are handled may factor into the degree to which samples represent the media from which they are collected. This, in turn, could affect the ability of decision makers to make accurate and timely decisions concerning the contamination status of the site. As appropriate, logbooks are assigned to, and maintained by, key field team personnel to record daily conditions/activities such as weather conditions, dates/times of significant events, level of personal protection equipment used, drilling activities, actual sample collection locations, photographs taken, problems encountered during field activities and corrective actions taken to overcome problems. Field log book assignments shall be recorded in the Site Logbook or other central file whose location is known to the FOL and the TOM. All field log book assignments, use, control, and archiving are governed by SOP CTO83-5 found in Appendix B of the WP. In addition to those requirements, the purpose of visits by site visitors shall be recorded. Together, field log books and sample documentation including COC forms provide a record that should allow a technically-qualified individual to reconstruct significant field activities for a particular day without resorting to memory. All sample records are eventually docketed into the Tetra Tech NUS, Inc. project central file. See Section 5.2.7 of the WP for additional information on sample documentation. Sample numbers (for samples submitted for chemical analysis) and respective COC numbers shall be recorded in the Field logbook.

A temperature blank shall be included in each cooler. By measuring the temperature of the temperature blank, the internal temperature of the cooler will be measured and recorded in the comments column of the COC form prior to sealing the cooler for shipment to the laboratory. Each cooler shall be taped shut with strapping tape in at least two places to prevent tampering. Custody tape shall be attached to span the cooler lid and sides so it must be broken to open the cooler. In accordance with NFESC guidelines (NFESC, 1996), samples for chemical analysis should be sent (for next-day receipt) to the laboratory within 24 hours of collection. For this investigation samples will be shipped to the laboratory within 24 hours of completing the sampling at a DE. This is necessary because random samples can only be selected for shipment according to grain size after all bore holes in the DE have been completed.

5.2 LABORATORY CUSTODY PROCEDURES

When samples are received by Laucks Testing Laboratories, Inc., 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 by measuring the temperature of the temperature blank. The temperature reading will be documented in the comments column of the COC form. In addition, the temperature reading will be recorded on the Supplemental Sample Receipt Log, as further explained below. The sample custodian will then sign the COC form and examine the contents of the cooler. Identification of broken sample containers or discrepancies between the COC form and sample labels will be recorded. A Laucks Testing Laboratories, Inc., CLP Sample Receipt Log and Supplemental Sample Receipt Log, as shown in Appendix 3 of SOP LTL-4002 (Appendix B), will be completed by Laucks Testing Laboratories, Inc. All problems or discrepancies noted during this process will be promptly reported to the TtNUS TOM. Samples will be logged into the laboratory information management system. Other pertinent issues relating to sample custody, such as interlaboratory COC procedures and specific procedures for sample handling, storage, dispersement for analysis, and remnant disposal, are presented in the laboratory SOPs included in Appendix B of this QAPP.

5.3 FINAL EVIDENCE FILES

The Administrative Record at NSWC Crane will be the repository for all documents which constitute evidence relevant to sampling and analysis activities as described in this QAPP. NSWC Crane will be the custodian of the evidence file and will maintain the contents of these files, 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 NSWC Crane Site Manager. The control file will include at a minimum:

- Field logbooks
- Field data and data deliverables
- Photographs and negatives
- Drawings
- Soil boring logs
- Laboratory data deliverables
- Data validation reports
- Data assessment reports

- Progress reports, Quality Assurance (QA) reports, interim project reports, etc.
- All custody documentation (tags, forms, airbills, etc.)

Upon completion of the contract, all files associated with this investigation will be maintained in the Administrative Record at NSWC Crane and will be available for inspection by the regulatory agencies for at least six years. Prior to disposal of all administrative records, the records will be offered to the U.S. EPA.

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. For this investigation, no field chemical measurements are intended; laboratory instrument calibration is outlined in Section 6.2.

6.1 FIELD INSTRUMENT CALIBRATION

No field chemical measurements are intended for this investigation and field instrument calibration is not required.

6.2 LABORATORY INSTRUMENT CALIBRATION

Calibration procedures for metals analyses by ICP/ES and ICP/MS begin with a periodic establishment of the useful linear response range followed by routine daily calibrations that consist of atomic mass calibrations (ICP/MS only), at least one blank and one calibration standard, an initial calibration verification, and continuing calibration verification standards/blanks, with each batch of samples analyzed. In all cases, an independently prepared standard (i.e., from a second source or a different lot number from the primary source) will be used as a calibration verification solution or as the MS spiking mix. Calibrations and associated documentation are required for all laboratory instruments. The documentation for calibrations performed in-house shall identify the person performing the calibration, the instrument being calibrated, the standards used for calibration and their concentration values or other pertinent calibration values, the source of the calibration standards, and the date of calibration. Certain instruments (e.g., balances) may be calibrated by a third party. In those cases the details of calibration as described above, and a certification of acceptable performance shall be obtained from the third party. The period during which the calibration is valid may appear in the calibration record or may be governed by standard operating procedure (SOP).

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

Calibration procedures, frequency requirements, acceptance criteria, and conditions that require recalibration are described for each analytical procedure in the applicable laboratory SOPs included in Appendix B.

7.0 ANALYTICAL AND MEASUREMENT PROCEDURES

All samples collected as part of this NSWC Crane background soils investigation will be analyzed by Laucks Testing Laboratories, Inc., 940 South Harney Street, Seattle, Washington 98108; (206) 767-5060; FAX (206) 767-5063. This laboratory has successfully completed the laboratory evaluation process required as part of the NFESC QA Program and described in the "Navy Installation Restoration Laboratory Quality Assurance Guide" (NFESC, 1996). Field measurements and analytical procedures are presented in detail in this section.

7.1 FIELD MEASUREMENT PROCEDURES

No field chemical measurements will be made as part of this investigation. Each sample collected, except surface soil samples, will be classified according to soil grain size.

7.2 LABORATORY ANALYTICAL PROCEDURES

Table 7-1 provides a summary of the laboratory analysis methods and associated laboratory SOPs to be used during this investigation. Laboratory SOPs for these analyses are included in Appendix B of the QAPP.

7.2.1 List of Project Target Compounds and Detection Limits

A complete list of the target analytes, project-specific risk-based target levels and soil-adjusted IDLs and RLs is provided in Section 1.4 of this QAPP. The IDLs provided for metals have been experimentally determined as described in Section 6.2.6 of Laucks Testing Laboratories, Inc., SOP LTL-1011, and adjusted for differences between water and soil matrices as explained in Section 1.4 of this QAPP. SOP LTL-1011 is based on the procedure for IDL determination as specified in the U.S. EPA CLP (ILM04.0; U.S. EPA, 1995). A digestate dilution factor of 5 (to limit the amount of dissolved solids entering the ICP/MS interface), and the professional judgment of experienced analysts, were considered when determining the soil-adjusted, sample-specific IDLs. All analyte concentrations will be reported as low as the soil-adjusted, sample-specific IDL; analyte concentrations less than the soil-adjusted, sample-specific IDL will be reported as the value of the soil-adjusted, sample-specific IDL with a "U" qualifier flag indicating that the analyte was not detected at that concentration. Analytes exceeding soil-adjusted, sample-specific IDLs but less than RLs will be considered to have been detected, but the results will be qualified by the laboratory with a "B" qualifier to indicate that the reported value is more uncertain than values reported at concentrations greater than the RL. Sample-specific RLs are based on the

corresponding IDLs with adjustments made to ensure that the precision and accuracy requirements of the method are attainable on a routine basis. RLs will be adjusted on a sample-by-sample basis, as necessary, based on dilutions, moisture content and sample volume. Method detection limits (MDLs) have not been determined in a soil matrix because, at the low concentrations detectable using ICP/MS analysis, the natural soil concentration variability would be inseparable from the analytical variability and MDLs could not be determined for naturally occurring metals.

7.2.2 List of Associated Quality Control Samples

In addition to the field QC samples presented in Section 3.0 of this QAPP, laboratory QC samples, including method blanks, preparation blanks, LCSs, etc., will be analyzed based upon method requirements. Laboratory QC samples are detailed in Section 8.0 of this QAPP. The analytical SOPs included in Appendix B address minimum QC requirements for each of the associated analytical methods.

TABLE 7-1

SUMMARY OF ANALYTICAL PROCEDURES
SOIL SAMPLES
NAVAL SURFACE WARFARE CENTER
CRANE, INDIANA

Analytical Parameter	Preparation Method[1]	Analytical Method[1]	Preparation/Analytical SOP(s) [2]
Al, Ca, Fe, K, Mg, Mn and Na - total	SW-846 3050B, with HNO_3 and H_2O_2 but **no** HCl	SW-846 6010B	LTL-7015/LTL-7202
TAL metals (except Al, Ca, Fe, Hg, Li, K, Mg, Mn, Na, Sb, Sn, Sr and Th) - total	SW-846 3050B, with HNO_3 and H_2O_2 but **no** HCl	SW-846 6020	LTL-7015/LTL-7105
Sb and Sn - total	SW-846 3050B, with Step 7.5 option using HNO_3 and HCl but **no** H_2O_2	SW-846 6020	LTL-7015/LTL-7202
Hg - total	SW-846 7471A	SW-846 7471A	LTL-7501/LTL-7501

1 U.S. EPA, 1986a. Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods. SW-846, 3rd Ed. and subsequent updates.
2 Laboratory SOPs are included in Appendix B.

8.0 INTERNAL QUALITY CONTROL CHECKS

No field measurement QC checks are required as no field measurements of chemical parameters are planned for this project. However, some field QC samples will be collected to evaluate overall data quality. This section provides information regarding internal QC checks for the field and the laboratory.

8.1 FIELD QUALITY CONTROL CHECKS

QC checks will be used to evaluate the quality of field operations. Collection of field QC samples will be in accordance with the procedures provided in Section 5.3 of the WP. Table 8-1 summarizes the minimum field QC sample collection requirements and associated corrective actions.

8.2 LABORATORY QUALITY CONTROL CHECKS

Laucks Testing Laboratories, Inc. operates a QC program that ensures the reliability and validity of the analyses performed at the laboratory. The laboratory's QA Plan describes the policies, organization, objectives, QC activities, and specific QA functions employed by the laboratory. All analytical procedures are documented in writing as SOPs. Each analytical SOP specifies minimum QC requirements for the procedure. As previously noted, SOPs for all analyses to be performed during this investigation are included in Appendix B of the QAPP. Table 7-1 provides a list of the SOPs associated with each analytical procedure. In addition, the laboratory maintains SOPs regarding general laboratory QA operations. Several of these SOPs, as applicable, are also included in Appendix B. The Table of Contents included in Appendix B provides a list of SOP titles and associated SOP numbers for all SOPs contained in the appendix.

Internal laboratory analytical QC requirements beyond those used for instrument calibration QC are briefly highlighted in the remainder of this section. Descriptions of the QC samples are provided in Section 3.7. Additional QC requirements which are specific to the NFESC QA Program, and are therefore requirements for this project, are also specified, as applicable, for each of the QC checks.

Laboratory method blanks acceptance criteria and corrective actions for non-compliant results are described in detail in SOPs LTL-7011, LTL-7105, LTL-7501 and LTL-7202 in Appendix B. Under no circumstances are laboratory method blank contaminant values subtracted from environmental sample analysis results.

Matrix spikes, to conform to NFESC requirements, will contain all the targeted analytes of interest. If the MS recovery is not within applicable control limits, the laboratory will assess the batch to determine whether the spike results are attributable to a matrix effect or are the result of other problems in the analytical process. Based on NFESC requirements, if all the batch QC elements which are not affected by the sample matrix are in control (e.g., method blank, LCS, calibration checks) and if there is no evidence that spiking was not properly performed, the poor spike recovery may be attributed to matrix effects. In this case, the associated data will be flagged, but re-preparation and re-analysis will not be required. If any of the batch QC elements which are not affected by the sample matrix are out of control, or if there is any evidence that spiking may have been improperly performed, the MS sample will be re-processed through the entire analytical sequence. If there is insufficient sample available, or if holding times have passed, the laboratory will flag the associated data. Details of noncompliant and laboratory duplicate results will be included in the sample delivery group (SDG) narrative.

Post Digestion Spikes will contain all target analytes of interest. They will be used to assist in assuring whether unacceptable MS recoveries are a result of matrix effects.

Laboratory control samples for metals analyses must contain all analytes of interest to be compliant with the NFESC QA Program. Laucks Testing Laboratories, Inc. will use LCSs purchased from an independent party and will apply the certified acceptance limits provided by that party as the control limits.

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

Laboratory duplicates will be used to assess laboratory precision. If RPD values exceed 50 percent, the analytical process will be investigated to assess whether the observed RPD value is an indication of a deficient analytical system or an indication of excess sample heterogeneity.

TABLE 8-1

FIELD QC SAMPLE COLLECTION FREQUENCIES
NAVAL SURFACE WARFARE CENTER
CRANE, INDIANA

QC Sample Type	Collection Frequency	Acceptance Limits	Corrective Action
Field Duplicate	1 per 10 investigative samples collected.	< 50% RPD for analytes with concentrations greater than 4*RL	Investigate to determine the cause of the large RPD. Qualify data according to data validation requirements if the observed RPD is correct.
Equipment Rinsate Blank	1 per 10 investigative samples collected, with a minimum of one per day of sampling, per sampling device/instrument. For pre-cleaned, dedicated, and/or disposable equipment (i.e., disposable plastic trowels, etc.), one rinsate blank will be collected and analyzed at a frequency of one per lot or "batch blank" for a specific equipment type.	< RL	Identify source of contamination, if possible. Qualify data according to validation criteria. Qualify use of background data set if contamination appears to have adversely affected the data usability.
Source Water Blank	1 per each source of water used for sampling equipment decontamination.	< RL	Identify source of contamination, if possible. Qualify data according to validation criteria. Qualify use of background data set if contamination appears to have adversely affected the data usability.
Ambient Condition Blanks	At discretion of FOL	< RL	Identify source of contamination, if possible. Qualify data according to validation criteria. Qualify use of background data set if contamination appears to have adversely affected the data usability.

9.0 DATA REDUCTION, VALIDATION, AND REPORTING

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

9.1 DATA REDUCTION

9.1.1 Field Data Reduction Procedures

No field environmental measurements will be made, hence data reduction is not required for field operations. However, all field logs containing observations will be inspected and approved by the FOL. All field observations will be recorded in the logs immediately after observations are made.

If errors are made in recording the observations, erroneous observations will be legibly crossed out using a single line, initialed, dated by the field member, and corrected in a space adjacent to the crossed-out entry. The FOL has responsibility to assure that errors are identified and assessed relative to the intent of the WP.

Errors judged to affect the utility of the sample results within the context of this investigation shall be brought to the immediate attention of the TOM.

9.1.2 Laboratory Data Reduction Procedures

Data reduction will be completed by Laucks Testing Laboratories, Inc. in accordance with the method-specific laboratory SOPs included in Appendix B. In addition, SOP LTL-1018 (provided in Appendix B) presents the procedures that will be used by Laucks Testing Laboratories, Inc., for review and approval of data.

Laboratory analytical data will be reported using standard concentration units to ensure comparability with previous analytical results. Soil sample results will be reported on a dry-weight basis in units of mg/kg.

9.2 DATA VALIDATION

Validation of field measurements and laboratory analytical data are presented in this section.

9.2.1 Procedures Used to Validate Field Data

Field measurements will not be subjected to a formal data validation process. Validation of field data will be limited to real time inspection by the FOL of observations relative to actual site conditions and activities. In addition, field technicians will ensure that the equipment used for sample collection is performing adequately via compliance with the applicable SOPs.

9.2.2 Procedures Used to Validate Laboratory Data

One hundred percent of the laboratory analytical data will be subjected to data validation to ensure that the data are of evidentiary quality. Validation of analytical data will be completed by the TtNUS Environmental Chemistry/Toxicology Department located in TtNUS's Pittsburgh office. Final review and approval of validation deliverables will be completed by the Department's Data Validation Coordinator.

Prior to statistical analyses, analytical results will be validated *versus* the applicable analytical methods, the SOPs included in Appendix B, and the requirements of this QAPP. Validation of these data will conform to the U.S. EPA Region 5 Standard Operating Procedures for Validation of CLP Inorganic Data (U.S. EPA Region 5, 1993b) and the NFG for Inorganic Data Review (U.S. EPA, 1994b) to the greatest extent practicable. Data validators shall review (on a timely basis) the chemical analytical data packages submitted by the laboratory. The data validators will check that the data were obtained using approved methodology, that the appropriate level of QC and reporting was conducted, and that the results are in conformance with QC criteria. On the basis of this information, 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 TOM. Data review will be extended beyond this routine validation by involving the project chemist, statistician, and risk assessor, as appropriate, to examine the data for anomalies. This additional review may result in more detailed inspections of the data to determine the cause of, and to rectify, individual anomalies. The impact of data qualifiers on data usability will also be assessed and any qualifications that are indicated during use of the background data shall be documented.

The data validation process will provide an estimate of the number of usable data points. This will be summarized by computing the number of data points that are rejected relative to the total number of data points for a given metal analyte in a given soil type.

9.3 DATA REPORTING

9.3.1 Field Data Reporting

Field data will be transferred from the site logbook or sample logsheets to the electronic database 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 Tetra Tech NUS central files upon completion of the field effort. Entry of these results in the database will require removal of these records from the files. Outcards (date, person, subject matter) will be used to document the removal of any such documentation from the files. After database entry is complete, all records will be copied for placement in Tetra Tech NUS central files; all original records will be sent to NSWC Crane for inclusion in the final evidence files, as described in Section 5.3.

9.3.2 Laboratory Data Reporting

Data reported by Laucks Testing Laboratories, Inc. for all analytical fractions will be in a CLP reporting format. All pertinent QC data including raw data and summary forms for blanks, standards analysis, calibration information, etc., will be provided for all analyses. Case narratives will be provided for each Sample Delivery Group (SDG). SOP LTL-4201 (Appendix B) provides further details regarding the information that will be included in CLP-type packages produced by Laucks Testing Laboratories, Inc.

Data will be handled electronically pursuant to the electronic deliverable requirements specified in TtNUS's Basic Ordering Agreement with analytical laboratories. This agreement requires the analytical laboratory to provide data in both hardcopy and electronic form. The database will include pertinent sampling information such as sample number, sampling date, and general location. Sample-specific quantitation limits will be reported for nondetected analytes. Units will be clearly summarized in the database and will conform to those identified in Section 9.1.2. The original electronic diskettes and data validation reports for this investigation will be maintained in the Administrative Record at NSWC Crane; copies will be maintained in Tetra Tech NUS central files.

Validation will be completed using the hard copy data. Upon completion of validation of a SDG and review by the Data Validation Coordinator, the validation qualifiers will be entered in the electronic database and will be subjected to independent review for accuracy. During this review process, the electronic database printout also will be compared with the hard copy data to ensure that the hard copy data and electronic data are consistent.

9.4 DATA ACQUISITION REQUIREMENTS AND DATA QUALITY MANAGEMENT

Several maps were used in selecting sampling points and in identifying site facilities and topography. Those maps are referenced in Sections 2.0 and 4.0 of the WP. In addition, a detailed evaluation of existing chemical data is provided in Section 3.0 of the WP.

9.5 DATA MANAGEMENT

Data management begins with the identification and collection of soil samples in accordance with the TtNUS Base-wide Background Soil Investigation Data Management Plan for NSWC Crane as provided in Appendix C. The samples are labeled and tagged, packaged for shipment, and shipped to the analytical laboratory in accordance with TtNUS SOPs as provided as attachments to the WP. The samples are received at the laboratory and analyzed, the analytical results are reported by the analyst along with quality control check data, and the data are reviewed within the laboratory, according to laboratory SOPs as provided in Appendix B. Data are then transmitted from the laboratory in both hardcopy and electronic formats according to laboratory SOPs as provided in Appendix B. The data are received at TtNUS, validated, analyzed, assessed, and ultimately archived, in accordance with the TtNUS Base-wide Background Soil Investigation Data Management Plan for NSWC Crane as provided in Appendix C. Data analyses will be performed by TtNUS as described in Section 6.0 of the WP.

10.0 PERFORMANCE AND SYSTEM AUDITS

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

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

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

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

10.1 FIELD PERFORMANCE AND SYSTEM AUDITS

This section presents the responsibilities, frequencies, and procedures associated with internal and external field performance and system audits.

10.1.1 Internal Field Audits

10.1.1.1 Internal Field Audit Responsibilities

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

10.1.1.2 Internal Field Audit Frequency

Internal field audits will be conducted at the beginning of the field program. This will allow for identification and correction of problems early in the program.

10.1.1.3 Internal Field Audit Procedure

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

- 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.) and field operations (sample COC, sample handling, etc.) to evaluate completeness and compliance with applicable SOPs.

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

- A formal post-audit debriefing will be conducted, and 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 TOM.

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

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

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

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

10.1.2 External Field Audits

10.1.2.1 External Field Audit Responsibilities

External field audits may be conducted by the IDEM, the U.S. EPA Region 5, or both.

10.1.2.2 External Field Audit Frequency

External field audits may be conducted at any time during field activities at the discretion of the IDEM and U.S. EPA Region 5. If an audit is to be conducted, scheduling should be coordinated through the QAM, to ensure that personnel and equipment are available as necessary. Personnel being audited may or may not be informed of the impending audit at the discretion and request of the auditing body.

10.1.2.3 Overview of the External Field Audit Process

External audit procedures are at the discretion of U.S. EPA Region 5 and the IDEM.

10.2 LABORATORY PERFORMANCE AND SYSTEMS AUDITS

This section presents the responsibilities, frequencies, and procedures associated with internal and external laboratory performance and system audits.

10.2.1 Internal Laboratory Audits

10.2.1.1 Internal Laboratory Audit Responsibilities

The QA/QC Officer or appropriate designee of the subcontracted laboratory performs routine internal audits of the laboratory. Internal laboratory audits also are conducted by the U.S. Navy. Tetra Tech NUS holds no responsibility for such audits. Performance and system audits of laboratories are coordinated through the NFESC by an independent QA contractor. It is the responsibility of the NFESC and its

contractor to ensure that the subcontracted laboratory complies with good laboratory practices and the general requirements of all analytical services provided by the laboratory.

10.2.1.2 Internal Laboratory Audit Frequency

Internal audits are performed bi-annually at Laucks Testing Laboratories, Inc., if no external audits are conducted. In addition, each laboratory department at Laucks Testing Laboratories, Inc., analyzes blind performance evaluation samples as described in SOP LTL-1009 (Appendix B). Data audits also are performed by the Laucks Testing Laboratory QA/QC Officer at least 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

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

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

10.2.2 <u>External Laboratory Audits</u>

10.2.2.1 External Laboratory Audit Responsibilities

External audits may be performed by the IDEM and U.S. EPA Region 5 at their discretion. Each laboratory also is involved in external audits and performance evaluation studies throughout the year, as required, to maintain certifications and/or approvals by other regulatory agencies or programs.

10.2.2.2 External Laboratory Audit Frequency

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

10.2.2.3 Overview of the External Laboratory Audit Process

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

11.0 PREVENTIVE MAINTENANCE PROCEDURES

Equipment used to collect samples will be maintained in accordance with the manufacturers' operation and maintenance manuals. Equipment and instruments will be calibrated in accordance with the procedures and at the frequency presented in Section 6.0 (Calibration Procedures and Frequency). Preventive maintenance for field and laboratory equipment are detailed in the remainder of this section.

11.1 FIELD EQUIPMENT PREVENTIVE MAINTENANCE

The TtNUS equipment manager and the equipment operator will be responsible for ensuring that equipment is operating properly prior to use and that routine maintenance is performed and documented. Any problems encountered while operating the instrument will be recorded in the field logbook, including a description of the symptoms and corrective actions taken. If problems with the equipment are detected and service is required, the equipment will be logged, tagged, and segregated from equipment in proper working order. Use of the equipment will not resume until the problem is corrected.

11.2 LABORATORY INSTRUMENT PREVENTIVE MAINTENANCE

Proper maintenance of laboratory instruments and equipment is essential. Depending on manufacturers' recommendations, maintenance intervals are established for each instrument. All instruments will be labeled with a model number and serial number, and a maintenance logbook will be maintained for each instrument. Personnel will be alert to the maintenance status of the equipment they are using at all times. Table 11-1 provides a summary of preventive maintenance procedures performed by Laucks Testing Laboratories, Inc., for key analytical instruments and equipment.

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

11.3 INSPECTION/ACCEPTANCE REQUIREMENTS FOR SUPPLIES AND CONSUMABLES

All field equipment shall be inspected prior to use to ensure that necessary parts are available. Most equipment planned for use in this project is simple with few to no moving parts. Therefore a visual

inspection prior to use shall be sufficient to ensure that the equipment is suitable for use. This visual inspection shall occur during mobilization and during each use by the person using the equipment.

Laboratory inspection and acceptance requirements are provided in Section 5 of the Laucks Testing Laboratories, Inc. Quality Assurance Plan, revision 8, dated December 1997. That plan presents the following specifications for inspection and acceptance of supplies and consumables:

- Requirements to follow individual SOP specifications for grades of chemicals necessary to achieve acceptable analytical performance. SOPs are required to detail the necessary grade of chemicals, including compressed gases.

- Requirements to obtain primary chemical standards from reliable sources that use calibrated glassware in the preparation of the standards. Emphasis is on obtaining NIST-traceable standards where possible.

- Storage of chemical standards in accordance with applicable SOPs and in a manner that preserves their integrity.

- Routine monitoring of de-ionized water and other solvents to ensure that analytical systems, samples and standards are not contaminated.

- Requirements to record the date received and the date opened on each container of chemical used for analysis.

TABLE 11-1

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

Instrument	Preventive Maintenance	Maintenance Frequency
ICP/MS	Clean or change air filters, change pump oil.	Semi-annually.
	Clean torch, replace nebulizer tips, replace pump tubing, replace injector, change cones.	As needed.
	Check mass calibration.	Every 2 weeks.
	Check sensitivity.	Daily.
ICP/ES	Service Intercooler.	Annually.
	Rinse and clean nebulizer cap and spray chamber.	Monthly or as needed.
	Clean torch, vacuum filters.	Bi-monthly.
	Profile instrument, examine autosampler tubing and replace as needed.	Daily.
	Empty rinse container, fill rinse water reservoir.	As needed.
Mercury Analyzer	Check and replace pump tubing, check and replace membrane, check and clean windows.	As needed.
Refrigerators	Monitor temperature	Daily
Ovens	Monitor temperature	Daily

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

Compliance with quantitative QC objectives for laboratory accuracy and precision as outlined in Tables 3-1 and 3-2 will be evaluated during data validation. No field data precision requirements are in effect for this investigation except to perform comparisons of field duplicate RPD values to laboratory duplicate RPD values. The validation process will be used to flag data whose quality indicators fall outside the QC acceptance limits. Compliance with completeness objectives for field and laboratory data will be computed manually. Sections 12.1 and 12.2 present equations to be used for computing precision and accuracy values, respectively. Section 12.3 describes the means for determining completeness. Section 12.4 addresses the overall data assessment process.

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

12.1 ACCURACY ASSESSMENT

Sample collection accuracy cannot be evaluated in this investigation because there is no standard by which to judge such accuracy. Sample analysis accuracy will be assessed through the use of matrix spikes (MSs), laboratory control samples (LCSs), calibration check standards, and blanks. Blanks will be used to infer the potential for positive biases due to contamination. To assure the accuracy of the analytical procedures, at least 1 of every 20 environmental samples will be spiked prior to digestion with a known amount of the analytes to be evaluated. The spiked sample will then be analyzed and the concentration of analyte observed in the spiked sample compared to the reported value of the analyte in the unspiked sample determines the percent recovery (%R) of the analyte. Control charts are plotted by the laboratory for each metal and kept on matrix- and analyte-specific bases. The %R for a spiked sample is calculated by using the following formula:

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

As discussed in Section 3.2.3, LCSs are also analyzed to assess accuracy. The %R calculation for LCSs is:

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

12.2 PRECISION ASSESSMENT

Variability of analyte concentrations in soil is the primary characteristic being investigated. Thus, there will be no attempt made to influence or control this parameter. However, laboratory duplicates will be compared to field duplicates to assess whether there is a difference in performance for the two types of samples. A duplicate field sample will be prepared and analyzed for every 10 environmental samples; a laboratory duplicate sample will be prepared for every 20 environmental samples collected. Duplicate field samples are prepared in the field by mixing an environmental sample to homogenize it, dividing it into two equally representative aliquots, and shipping the aliquots in separate containers to the laboratory. Duplicate laboratory samples are prepared by the laboratory by removing two aliquots of material from a mixed sample. The RPD between a sample (Sample 1) and its duplicate (Sample 2) is calculated according to the following formula and is plotted:

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

12.3 COMPLETENESS ASSESSMENT

Completeness for this project will be determined based on the number of sample results for each metal and each soil type that are usable as determined through data validation and data assessment. Data values rejected during data validation (indicated by an “R” flag) will be considered unusable unless additional review and documentation by one or more technical team members demonstrates that the rejection is erroneous. To monitor completeness, the number of usable, valid results for each soil type and analyte will be counted and compared to the completeness objectives in Sections 3.3.2 and 3.3.3.

12.4 DATA ASSESSMENT

The assessment of data obtained from this background investigation is a critical part of determining what the next step in data collection and decision making should be. It must be determined if the data are of appropriate type, quality, quantity and representativeness to support the project objectives. The effect of the loss of data deemed unacceptable for use, for whatever reason, must be discussed.

The field and laboratory data collected during the investigation will be used to characterize the background metal concentrations in the selected soil types at NSWC Crane as described in Section 5.2 of the WP. Those characterizations are designed to qualitatively and quantitatively assess the data on an investigation-wide basis. The results for each metal in each soil type will be compared to determine whether the concentration distributions and spatial distributions differ significantly as determined through statistical testing and best professional judgment. The QC results associated with each analytical parameter for each matrix will be compared to the objectives presented in Sections 3.1 though 3.5 of this QAPP. At a minimum, the following issues to be considered in this assessment are:

- Deviations, if any, from the field sampling SOPs
- Deviations, if any, from the laboratory analytical SOPs
- Deviations, if any, from the QAPP
- Deviations, if any, from the WP
- Deviations, if any, from the data validation process
- Identification and explanation of elevated reporting limits
- Identification of unusable data (i.e., data qualified as “R”)
- Validity of background investigation assumptions
- Adherence to investigation objectives and decision rules
- Completion of corrective actions
- Effects of deviations from planned procedures and processes on the interpretation and utility of the data
- The existence of remaining data gaps
- If necessary, revision of investigation objectives in anticipation of additional data collection

With the following exceptions, National Functional Guidelines (NFG) acceptance criteria must be satisfied or the data will be qualified for use according to NFG rules (U.S. EPA, 1994b):

- Failure to meet bias acceptance criteria may be allowed on a case by case basis with input from the technical team and with approval from the TOM. For example, failure to meet acceptance criteria because of excess data scatter rather than an analytical bias may be acceptable, although it might indicate a need to collect additional samples for inclusion in the background data set.

- Duplicate sample acceptance criteria will not apply because the primary objective of this investigation is to characterize the soil concentration distributions. Further, on the scale at which soil samples are subsampled in the laboratory, there can be no real duplicate analysis of soil samples unless they are

subjected to particle size reduction and homogenization. Particle size reduction was considered but rejected for this investigation (see Section 1.1.3).

Data qualified during data validation will be reviewed to determine the impact of the qualification(s). Rejected values, in particular, may be reviewed to determine if the cause of the rejection status is pervasive. J-qualified values will be evaluated to assess whether the qualification indicates only a greater than usual uncertainty or a potential bias.

It is acknowledged that bias estimates may not be accurately quantifiable with limited amounts of QC data and it is not the primary objective of this investigation to identify or quantify biases in sample collection, handling, preparation, and analysis. All determinations of detectable difference among data sets will assume no relative bias between sample collection, handling, and analysis processes used in this investigation and the same processes used in future environmental investigations. However, during the course of data analysis and assessment, biases will potentially be identified and quantified as measures of data comparability for consideration during future background data comparisons. Biases, or the potential for biases, will be estimated using the analyte recovery data from MSs, LCSs, calibration check standards, and blanks. It is noteworthy that background comparisons made using data generated by identical sampling and analysis methods incorporate similar biases and are directly comparable. Verifiable biases will be documented.

One consideration of particular importance is the consideration of sufficient sample data. If fewer than five results are available for a target metal in a particular soil type, consideration will be given to the costs of collecting additional background data and to the costs of collecting additional samples in SWMU investigations. Probability tables, based on the actual grain size classifications obtained in this investigation, will be generated to show the number of additional samples that would have to be collected to obtain the target number of five samples within a soil type. Based on those estimates, costs can be estimated.

Detail of the process used to evaluate data relative to project objectives is explained in Section 6.2 of the WP. That process includes the statistical tests used to compare data sets, and to identify and evaluate potential outliers. All statistical testing will be based on standard statistical practices and will be fully documented.

The TOM shall bear ultimate responsibility to ensure that data are evaluated in a manner consistent with project objectives. All data, including statistical outliers, will be retained as part of the final record even though they may not be used in decision making. If any data indicate that a sample location has been the

location of an actual or potential release, the validity of the data for inclusion in the background data set will be re-evaluated.

13.0 CORRECTIVE ACTION

Under the TtNUS QA/QC program, it is required that any and all personnel noting conditions adverse to quality report these conditions immediately to the TOM and QAM. These parties, in turn, are charged with performing root-cause analyses and implementing appropriate corrective action in a timely manner. 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 product quality is not compromised. The need for corrective action may arise based on deviations from project plans and procedures, adverse field conditions, or other unforeseen circumstances. Corrective action needs may become apparent during the performance of daily work tasks or as a consequence of internal or external field audits.

Corrective action may include resampling and may involve amending previously approved field procedures. Minor modifications to field activities, such as the collection of additional samples, will be initiated at the discretion of the FOL, subject to onsite approval by NSWC Crane personnel. Major modifications, such as the elimination of a sampling point or other situations that affect compliance with or achievement of DQOs, must be approved and documented via a Field Task Modification Request (FTMR) as described in Section 5.2.7 of the WP. Approval of the corrective action will be obtained by the U.S. Navy (in conjunction with U.S. EPA Region 5 and IDEM). The FOL is responsible for initiating FTMRs; a FTMR will be prepared for all deviations from the project plan documents, as applicable. An example of a FTMR is provided in Appendix A of the WP. Copies of all FTMRs will be maintained with the onsite project planning documents and will be placed in the final evidence file.

13.2 LABORATORY CORRECTIVE ACTION

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

- 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 %Rs or in duplicate RPDs
- There is an unexplained change in compound detection capability
- Inquiries concerning data quality are received
- Deficiencies are detected by laboratory QA staff audits or from PE sample test results

Any corrective action taken above the analyst level that cannot be performed immediately at the instrument will be documented. Corrective actions typically are documented for out-of-control situations on a Corrective Action Form or an Out-of-Control Event Form. Copies of the forms used by Laucks Testing Laboratories, Inc., are included as Appendices 1 and 3 of SOP LTL-1008, which is in Appendix B 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.

Laboratory corrective actions must be documented and included as part of the Final Evidence File. Major corrective actions that do not bring DQO-related non-conformances into conformance with project DQOs shall be identified to the TtNUS TOM who will advise all levels of project management in accordance with Section 14.0 of this QAPP.

13.3 CORRECTIVE ACTION DURING DATA VALIDATION AND DATA ASSESSMENT

The need for corrective action may become apparent during data validation, interpretation, or presentation activities. The performance of rework (i.e., resampling or reanalysis), the institution of a change in work procedures, or the provision of additional/refresher training are possible corrective actions relevant to data evaluation activities. The TOM will be responsible for approving the implementation of a corrective action and ensuring that it is documented appropriately.

14.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

QA reports to management will be provided in four primary formats during the course of this investigation: data validation reports, reports summarizing accomplishments and QA/QC issues during the field investigation, project-wide progress reports, and laboratory QA reports.

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

The FOL will provide the TOM with daily oral field progress reports during the course of the sampling event. These reports will explain accomplishments, deviations from the WP, upcoming activities, and a QA summary. The TOM provides a monthly progress report to the Navy which addresses the project budget, schedule, accomplishments, planned activities, and QA/QC issues and intended corrective actions.

The subcontracted analytical laboratories will provide a QA report to TtNUS if QC limits are updated or if other significant plan deviations resulted from unanticipated circumstances. Since IDLs/RLs, as applicable, will be included in the analytical data packages for NSWC Crane samples, it is not necessary for the laboratories to include updated IDLs/RLs in their QA reports unless the updates result in RLs which exceed risk-based target levels.

The frequencies of report generation, report content, report preparer, and report recipient(s) are summarized in Table 14-1.

TABLE 14-1

SUMMARY OF REPORTS
NAVAL SURFACE WARFARE CENTER
CRANE, INDIANA

Report	Content	Preparer	Frequency of Submittal	Recipient(s)
Data Validation Report	All major and minor laboratory noncompliances as well as noted sample matrix effects	Data Validation Coordinator or designee	Per SDG	TOM, project file
Major Analysis Problem Identification Report (internal memorandum)	Notification of persistent or major problems with analytical laboratory performance. Summary of the noncompliance(s), a synopsis of the impact on the project, and recommendations regarding corrective action and compensation adjustments	Data Validation Coordinator or designee	When persistent analysis problems are detected	TOM, QAM, Technical Program Manager, Deputy Program Manager, and Laboratory Services Coordinator, project file
Project Monthly Progress Report	Summary of the project budget, schedule, accomplishments, planned activities, and QA/QC issues and intended corrective actions.	TOM	Monthly for duration of project	Navy, project file
Field Progress Reports	Explain accomplishments, deviations from the FSP, upcoming activities, and a QA summary.	FOL	Daily, oral	TOM
Laboratory QA Report	Summary of updated QC limits or significant deviations from planned activities/performance.	Laucks Testing Laboratories, Inc.	When QC limits are updated or when other significant plan deviations result from unanticipated circumstances	Tetra Tech NUS, project file; US EPA Region 5, if changes in IDLs/RLs cause them to exceed risk-based target levels or if QAPP deviations impact DQOs
Any QA Report	See descriptions above	Tetra Tech NUS	Within 48 hrs of request from US EPA Region 5	US EPA Region 5

SDG = Sample delivery group
TOM = Task order manager
QAM = Quality Assurance Manager
IDL = Instrument Detection Limit
RC = Reporting Limit
FOL = Field Operations Leader

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APPENDIX A

HUMAN HEALTH AND ECOLOGICAL RISK-BASED CRITERIA

TABLE A-1

HUMAN HEALTH AND ECOLOGICAL RISK-BASED CRITERIA

Chemical	U.S. EPA SSL[1]			Region 9 PRG[2]		IDEM Tier 1 Default Cleanup Level[3]			
						Residential Land Use		Non-Residential Land Use	
	Ingestion (mg/kg)	Inhalation (mg/kg)	Migration to Ground Water (mg/kg)	Residential Land Use (mg/kg)	Industrial Land Use (mg/kg)	Surface Soil (mg/kg)	Subsurface Soil (mg/kg)	Surface Soil (mg/kg)	Subsurface Soil (mg/kg)
Aluminum				75000	100000				
Antimony	31		0.3	30	750				
Arsenic	0.4	750	1	0.38	3	3.9	29.2	19.6	29.2
Barium	5500	690000	82	5200	100000	1000	1000	1000	1000
Beryllium	0.1	1300	3	0.14	1.2	1.87	63.2	10.1	63.2
Cadmium	78	1800	0.4	37	930	24	7.52	1000	154
Calcium									
Chromium, Total	390	270	2	30[5]	64[5]	1000	38.4	1000	196
Cobalt				3300	29000				
Copper				2800	70000				
Iron				22000	100000				
Lead	400[6]			400[6]	1000[6]				
Lithium				1,500	37,000				
Magnesium									
Manganese				3100	45000				
Mercury	23	10	0.1	22	560	54.7	2.09	147	32
Nickel	1600	13000	7	1500	37000	1000	130	1000	1000
Potassium									
Selenium	390		0.3	370	9400	1000	5.2	1000	53.1
Silver	390		2	370	9400	1000	31	1000	86.9
Sodium									
Strontium				45,000	100,000				
Thallium			0.04	6[7]	150[7]	1000	2.85	1000	1000
Thorium									
Tin				45000	100000				
Vanadium	550		300	520	13000				
Zinc	23000		620	22000	100000	1000	1000	1000	1000

1 U.S. EPA Soil Screening Levels. (Soil Screening Guidance Technical Background Document. EPA/540/R-95/128. Office of Solid Waste and Emergency Response, Washington, D.C. Directive 9355.4-17A, May 1996.) For migration to ground water, values associated with a dilution and attenuation factor (DAF) of 1 are used.
2 U.S. EPA Region 9 Preliminary Remediation Goal. (U.S. EPA Region 9, May 1998.)
3 Risk-Integrated System of Cleanups. Indiana Department of Environmental Management. October 21, 1997.
4 U.S. EPA Region 5 Ecological Data Quality Levels (April 1998).

APPENDIX B

STANDARD OPERATING PROCEDURES

APPENDIX B

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SOP Number	Revision	Title
Laucks Testing Laboratories, Inc. SOPs:		
• LTL-1001	3	Elements of SOP and Method Formats
• LTL-1002	3	Document Tracking and Control
• LTL-1003	2	Chain-of-Custody and Documentation Procedures
• LTL-1004	3	Documentation of Analyst Competence and Training
• LTL-1005	4	Analytical Balances
• LTL-1006	2	Refrigerator, Freezer, and Oven Thermometer Calibration and Maintenance
• LTL-1007	2	Use of Instrument Records and Logbooks
• LTL-1008	5	QC Corrective Action
• LTL-1009	2	Blind Spike Program
• LTL-1011	4	Procedures for the Determination and Reporting of Detection Limits, Reporting Limits, Precision and Accuracy Studies, and Control Limits
• LTL-1013	3	Preparation, Storage, Shelf Life and Traceability Documentation of Standards and Reference Materials
• LTL-1017	1	Internal Audit Procedures
• LTL-1018	0	Overview of Review and Approval Practices for Validatable Data Packages
• LTL-1019	1	Controlling, Maintaining, and Monitoring Laboratory Logbooks
• LTL-2001	5	Waste Segregation and Disposal
• LTL-4002	5	Electronic Sample Entry and Log-In
• LTL-4103	0	Reviewing a Sample Delivery Group (SDG) and Updating Projqc in the LIMS
• LTL-4201	0	Package Deliverables for all Reporting Levels
• LTL-7003	2	Inorganic Glass Cleaning Procedure
• LTL-7015	2	Acid Digestion of Sediments, Sludges, and Soils by SW846 Method 3050B
• LTL-7105	0	Method Protocols for the Analysis of Metals by ICP Using SW-846 6010B
• LTL-7202	3	Metals Analysis Using Inductively Coupled Plasma - Mass Spectrometry (ICP/MS) SW846 Method 6020
• LTL-7501	1	Operation of the PE FIMS 400 for the Analysis of Hg

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP No. **LTL-1001**

Previous SOP No. **LTL-0001**

Title: **Elements of SOP and Method Formats**
Rev: 3

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

UNCONTROLLED

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #~~LTL-0001~~ 12/26/95
LTL-1001

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

Revision history:

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

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

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

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

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1. Introduction and Scope

1.1. The purpose of this SOP is to define the process of creating SOPs and Methods. SOPs are considered to be administrative and other non-analytical tasks. Methods are considered to be technical analytical procedures and are generally derived from EPA or other analysis methods. Both have similarities and differences in structure and necessary elements. This document assumes that the user either has some knowledge of the word processor being used or can figure out how to perform the basic operations necessary.

2. Procedures

2.1. Word Processing Formats

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

2.2. Initiating an SOP or Method

2.2.1. Prior to creating a new SOP or Method or revising an existing document, the prospective author or supervisor should first complete a Document Control Form as specified in the document control SOP, LTL-0054. This form can be obtained from the QC Department.

2.2.2. SOPs are considered to be administrative and other non-analytical tasks. Methods are considered to be technical analytical procedures and are generally derived from EPA or other analysis methods. At the time of initiation of a new document, an SOP or Method number will be assigned by the QC Officer or designee. SOPs will be prefixed with the letters LTL- (such as LTL-0001). Methods will be prefixed with the letters LX- (such as LX-0123).

2.2.3. If a revision of a previous document is being undertaken, the SOP or Method number will remain the same but the revision number will be incremented. Revision numbers of new documents will automatically be assigned as 1.0 with subsequent revisions generally being incremented by 1 (2.0, 3.0, etc.)

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

from the QC Department in order to ensure that the latest version is being used. These are meant for guidance only and changes to the formats will be allowed if they present a more complete and accurate account of method performance. Electronic copies may be obtained in several ways.

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

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

2.2.7. On some computers, the template is stored as a template file. It may be easily accessed by choosing [File][New] and the template name (SOPhead, Iorgtemp or Orgtemp). All of these templates are written in Word. These templates were written primarily as guidelines for comprehensive writing of an SOP or method. While it is entirely up to the author to change any part of one of these templates to suit the specific procedure, these elements will be looked at in the review process and must be included unless they are inappropriate to the procedure being described.

2.2.8. SOPhead is the very general SOP template. This template is NOT to be used for analytical methods as it does not contain all of the necessary elements of a method. Specific elements of a method are outlined below and in the method templates Iorgtemp and Orgtemp.

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

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

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

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

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

does not mean that text which is black cannot be modified or even deleted if it is not pertinent to the analysis in question.

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

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

3. Elements Of An SOP/Method

3.1. Elements

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

3.1.2. Methods contain the appropriate cover and header information, signature sheet, introduction and scope, equipment, reagents, specific operating procedures, calibration and quality control (including corrective actions), and any appropriate appendices. They should also include data package assembly information and run sequences. Required appendices include preparation of standard solutions, a Method QC Table and a procedural flow chart.

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

The laboratory name
The SOP/Method number (assigned by QC Officer)
The title of the SOP including EPA method number reference when appropriate
The revision history (revision number and date of approved revision)
Signature of Author and date signed

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

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

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

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

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

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

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

3.1.10. A brief description of the process delineated in the rest of the text. Where the process described varies from an accepted methodology (such as SW 846 or CLP), the variations should be clearly depicted in this section.

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

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

3.1.13. All SOPs/Methods contain a section called Equipment List and Standards (and/or reagents). All equipment and solutions necessary to complete the process described should be outlined in this section.

3.1.14. All SOPs/Methods contain a section called Safety Precautions and Waste Disposal. Any potential safety hazards should be depicted here as well as all waste disposal processes that may be entailed. If disposal involves pouring the waste into a collection container, that is all the description that is needed. The SOP then only need reference the waste disposal SOP (LTL-0032) for final disposal.

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

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

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

3.1.18. Finally, SOPs and Methods, while not always required, will often contain Appendices. Two specific appendices common to most methods are a Quality Control Summary Table and

a flow chart which depicts the basic steps involved in completing the process in the routine order and which includes the evaluation of successful completion of that process (i.e. "Is the QC in Control? If so, report the data. If not, what next?")

3.2. Saving the Document

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

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

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

3.2.4. Note that in either word-processing format, if you want to save the document to any other drive or directory than it was called from, you will have to specify that path.

3.3. After Completion of the Draft SOP

3.3.1. After the document has been written to the satisfaction of the author, it should be passed to the QC Officer for review and distribution. An unapproved SOP or Method document is not considered official.

Appendix I

SOP Template (WORD)

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-0000

Title: (Place title here)

Revision history:

Number	Date

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

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

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

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

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

Title: (Place title here)

Operator's signature page

Signature	Printed Name	Date

Signature page, for department manager's files

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1. Introduction and Scope

1.1. Method Description

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

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

1.2. Definition of Terms

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

2. Equipment List and Standards

2.1. Equipment

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

2.2. Reagents

2.3. Standards

3. Safety precautions and Waste Disposal

3.1. Safety Precautions

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

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

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

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

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

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

3.2. Waste Disposal

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

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

4. Calibration and Quality Control

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

5. Operation procedures

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

6. Reports

6.1. Data Package Organization

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

Appendix I

Flow Chart

Appendix II

(Title here)

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

Appendix II (WORD)

Inorganics Method Template

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LX-0000

Title: (Place title here)

Revision history:
Number Date

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

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

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

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

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

Title: (Place title here)

Operator's signature page

Signature	Printed Name	Date

Signature page, for department manager's files

1. Introduction and Scope

1.1 Method Description

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

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

1.2 Sample Collection, Sample Storage, Holding Times

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

1.3 Definition of Terms

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

Blank spike - A background free matrix (DIW for water, clean sand for soils/sediments) to which known amounts of target analytes are added each time samples are prepared. Blank spikes are required on all HAZWRAP and NEESA work. Note that an LCS or SRM (see below) will substitute as a blank spike for most inorganic analyses. In the context of this SOP, a blank spike is the same as a QC check standard. See also QC check standard.

CCV - Continuing calibration verification. - This is the same acronym used in the CLP program. This is a standard analyzed at some prescribed frequency (almost always after every 10 samples) during the analysis sequence to determine whether the instrument or system has remained in calibration.

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

Equipment List and Standards

Instrument

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

Standards

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

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

Safety precautions and Waste Disposal

Safety Precautions

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

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

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

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

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

Waste Disposal

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

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

Calibration and Quality Control

Method Detection Limit Study

Prior to the analysis of any samples, it is necessary to establish method detection limits. This procedure is fully described in Laucks SOP LTL-0027. Briefly, it involves the analysis of 7 replicate samples spiked at a concentration near the anticipated method detection limit. A Student's T-test is then applied to these measured values to calculate the MDL.

Initial Multi-Point Calibration

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

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

Criteria

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

Corrective action

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

Initial Calibration Verification

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

Criteria

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

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

Corrective action

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

Initial Calibration Blank

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

Corrective action

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

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

Method Detection Limit Standard

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

Corrective Action

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

Continuing Calibration Verification (CCV) and Blank (CCB)

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

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

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

Corrective action

If CCV limits are exceeded, check calculations or perform instrument maintenance. Recalibrate and reanalyze. No sample results may be reported that are not bracketed by a successful calibration and a CCV which is in control or by preceding and following CCVs which are within limits.

If the initial CCB contains target analyte levels above twice the detection limit, the system is out of control. The source of contamination must be identified and corrected and the affected samples re-analyzed. As with the CCVs, no sample results may be reported that are not bracketed by a successful initial and continuing calibration blank which are in control or by preceding and following CCBs which are within limits.

Method Blanks

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

Corrective action

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

Method Blank Spikes

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

Corrective action

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

Matrix Spike

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

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

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

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

Corrective action

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

Matrix Spike Duplicate

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

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

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

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

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

Sample Duplicate

Criteria

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

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

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

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

Corrective action

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

control and no analyte of interest was detected in any of the samples, no immediate action will be taken on that sample set. If integrity of reported sample values is in doubt, re-analysis may be called for. Corrective actions should be discussed with the Quality Control Officer.

Operation procedures

Sample Analysis

Analysis sequence

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

Instrumental Conditions

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

Analytical Operation

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

Compound Quantification

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

Reports

Data Packet Organization

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

Quality Control Reports

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

Add in any other specific stuff here.

Sample Result Reports

Data Qualifying Flags

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

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

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

Control Chart(s)

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

Appendix III

Organics Method Template (WORD)

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LX-????

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

Revision history:

Number	**Date**
draft 1.0	08/31/95

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

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

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1. Introduction and Scope

1.1. Method Description

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

Method Criteria	Laucks Variations

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

1.2. Sample Collection, Sample Storage, Holding Times

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

1.3. Definition of Terms

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

1.3.2.	Batch Identifier	A number given to each preparation or analysis group which uniquely identifies that batch. This number is generally the blank ID for preparation batches and either a sequence number for organic analyses or an analysis number which is similar to the blank ID, only preceded by an "A" rather than a "B" for inorganic batches. The preparation batch IDs are discussed in other documentation. You should expand upon how the batch is numbered for your particular application (i.e. B or A, mmddyy, analysis ID, matrix, sequence #).
1.3.3.	Blank spike	A background free matrix (DIW for water, clean sand for soils/sediments) to which known amounts of target analytes and surrogates are added each time sample extracts are prepared. Blank spikes are required on all HAZWRAP and NEESA work. In the context of this SOP, a blank spike is the same as a QC check standard. See also QC check standard.
1.3.4.	CCV	Continuing calibration verification. This is the same acronym used in the CLP program. This is a standard analyzed at some prescribed frequency during the analysis sequence to verify that the instrument has remained in calibration.
1.3.5.	CF	Calibration factor. The ratio of analyte instrument response to nanograms injected. This term is defined in the same way in both the CLP contract and SW 846.
1.3.6.	CLP	Contract Laboratory Program. The USEPA program that contracts with laboratories to provide laboratory services. The term has come to mean a much broader set of methods and deliverables. In the context of this SOP, CLP means procedures or operations which are detailed in the CLP contract and which are extended to a broader working definition.
1.3.7.	Corr Coef, CC	Correlation coefficient. A measure of the "goodness of fit" of a set of data to a regression model. The closer the value is to 1, the higher the degree of confidence in the correlation.
1.3.8.	DIW	Deionized water. Lab reagent water. Organic-free water. Since the systems used to provide DIW at Laucks all contain carbon polishing filters, they are capable of providing organic-free water for use in method blanks and method blank spikes.

1.3.9. IBLK	Instrument blank. This term is borrowed from CLP. Blank solvent containing the method surrogates is injected into the instrument to monitor for carry over between sample extract injections.
1.3.10. ICV	Initial calibration verification. This term is borrowed from the CLP GC/MS protocol. It is a standard which is injected at the start of each QC period that is compared to the initial multi-point calibration to determine whether the instrument is still in calibration.
1.3.11. IDL	Instrument detection limit. The lowest concentration of a target analyte that will yield a signal:noise ratio of at least 3x. Used as a starting point for selecting MDL study spiking levels.
1.3.12. MDL	Method detection limit. The lowest concentration in a sample which will yield a positive result that is greater than zero at a known level of confidence. MDLs are empirically determined by Laucks.
1.3.13. MDL standard	Method detection limit standard. A standard prepared so that the concentrations of the target analytes are in the range of 1x to 4x the empirically determined MDLs on an extract/digest basis. This standard is used to verify that the instrument is capable of detecting the target analytes on an ongoing basis.
1.3.14. PQL or Reporting Limit	Practical Quantitation Limit or Reporting Limit- The value used when reporting a non-detect. It may be administratively, empirically or contractually set.
1.3.15. QC check standard	Quality control check standard. Referred to in this SOP as a blank spike. A QC check standard is a requirement of SW 846 method 8000 and is used to determine whether the analytical system is in control if MS/MSD recoveries are out of control. See also blank spike.
1.3.16. QC period	Quality control period. An analysis sequence initiated by the analysis of one or more standards, followed by sample extracts/digests, and terminated with a standard analysis. A QC period can be open-ended chronologically, but calibration verification must be documented using the procedures in this SOP.

Appendix I

Standard Solutions

Appendix II

QC Summary Table

Laucks Testing Laboratories

Method _____ QA Requirements and Corrective Actions

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

Appendix III

Flow Chart

Appendix IV

(Title here)

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

Appendix III

Organics Method Template (WORD)

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

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

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

1.3.18. RSD or %RSD — Relative standard deviation or percent relative standard deviation. The ratio of the standard deviation of a set of values to the mean of the set of values expressed as a percentage. A measure of the similarity of the values one to another.

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

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

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

1.3.22. SRM — Standard Reference Material - A material containing known quantities of target analytes in a homogeneous matrix which approximates the matrix of the samples being analyzed. It is used to establish that the analytical process is in control.

1.4. Note on Using Spreadsheets for Standard Deviation Calculations

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

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

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

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

2. Equipment List and Standards

2.1. Chromatographic System

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

2.2. Standards

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

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

3. Safety precautions and Waste Disposal

3.1. Routine Safety Precautions

3.1.1. All standards and sample extracts should be handled as if they contain hazardous substances.

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

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

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

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

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

3.2. Waste disposal

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

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

4. Calibration and Quality Control

4.1. Method Detection Limit Study

4.1.1. Prior to the analysis of any samples, it is necessary to establish method detection limits. This procedure is fully described in Laucks SOP LTL-0027. Briefly, it involves the analysis of 7 replicate samples spiked at a concentration near the anticipated method detection limit. A Student's T-test is then applied to these measured values to calculate the MDL.

4.2. Method Validation

4.2.1. Prior to the analysis of any samples, it is necessary to validate the method. In many cases, the data from the MDL study may be sufficient for method validation. If the RSDs are too high, it will be necessary to perform a method validation study. A method validation study is performed in a similar manner to an MDL study with the exception that a minimum of 4 replicates are required and the concentration levels are typically higher.

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

4.3. Retention Time Windows

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

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

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

4.4. Initial Multi-Point Calibration

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

4.5. External Standard Calibration

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

4.5.2. CFs are calculated using the equation:

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

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

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

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

4.5.1. Corrective action

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

4.6. Internal Standard Calibration

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

4.6.2. RRFs are calculated using the equation:

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

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

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

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

4.6.1. Corrective action

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

4.7. Initial Calibration Verification

4.7.1. Concentration and/or CF Criteria

4.7.1.1. At the beginning of an analysis sequence analyze a mid-range calibration standard. The computed calibration factor (CF) or concentration measurement must meet the criteria detailed below.

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

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

where:

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

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

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

where:

C_t = True concentration
C_i = Measured concentration

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

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

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

4.7.2. Corrective action

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

4.8. Chromatographic Resolution

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

4.8.2. Criteria

4.8.2.1. Detail any resolution criteria here.

4.8.3. Corrective action

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

4.9. Updating Retention Time Windows

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

4.10. Instrument Blank

4.10.1. Criteria

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

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

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

4.10.2. Corrective action

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

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

4.11. Method Detection Limit Standard

4.11.1. Criteria

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

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

4.11.2. Corrective action

4.11.2.1. Perform system maintenance before analyzing any sample extracts.

4.12. Continuing Calibration Verification

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

4.12.1. Criteria

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

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

where:

CF_i = CF from ICV standard

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

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

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

4.12.2. Corrective action

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

4.13. Method Blanks

4.13.1. Criteria

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

4.13.2. Corrective action

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

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

4.14. Method Blank Spikes

4.14.1. Criteria

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

4.14.2. Corrective action

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

4.15. Matrix Spike

4.15.1. Criteria

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

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

where:

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

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

4.15.2. Corrective action

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

4.16.Matrix Spike Duplicate

4.16.1. Criteria

4.16.1.1. The compound recovery criteria are identical to those for the matrix spike sample. In addition, the matrix spike duplicate is used measure method precision. This is done by computing the relative percent difference (RPD) between the matrix spike and matrix spike duplicate recovery values. This calculation is as follows:

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

where:

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

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

4.16.2. Corrective action

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

4.17. Sample Duplicate

4.17.1. Criteria

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

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

where:

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

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

4.17.2. Corrective Action

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

4.18. Surrogate Recovery

4.18.1. Criteria

4.18.1.1. Surrogates are chemically similar compounds added to every sample, method blank, and QC sample prior to sample processing. They are used to monitor for potential sample processing errors and matrix effects. Surrogate compound recoveries are calculated as follows:

$$\% \text{ recovery} = \frac{S_m \times 100}{S_a}$$

where:

S_m = concentration of surrogate measured in extract

S_a = concentration of surrogate added

4.18.1.2. Detailed surrogate recovery control limits are tabulated in the Control Limits Catalog and/or the QC database, QC_DB.

4.18.2. Corrective Action

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

4.18.2.2. Low surrogate recoveries in the method blank may require that all the samples in the associated batch be re-extracted and re-analyzed. In any case, it is imperative to identify the problem associated with low recovery so that it can be corrected. It is a requirement that all out of control surrogate recoveries and the corrective action taken be discussed in the narrative.

5. Operation procedures

5.1. Chromatographic Conditions

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

5.2. Sample Analysis

5.2.1. Analysis sequence

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

5.2.2. Compound Identification

5.2.2.1. Compounds are tentatively identified if a peak elutes in the retention time window characteristic of that compound on the primary column. To confirm the presence of that compound in the sample extract, the peak must also elute in its characteristic retention time window on a second column. Retention time windows are established as previously described and are updated each QC period. Compounds can only be identified if the ICV and CCV criteria previously detailed are strictly adhered to.

5.2.2.2. The experienced analyst's judgment weighs heavily in evaluating chromatograms for compound identification. For instance, the retention times of surrogate compounds may

be outside their expected windows due to sample matrix effects. The analyst may decide to re-adjust the target analyte's retention time windows on an ad ad hoc basis based on such an observed shift. If the data are processed using CHRO, the software allows the operator to increase the retention time window half-width beyond the method- specified width. This can occur only on a sample-specific basis and is used when the analyst examining the data suspects that a retention time shift has occurred. If this is done, it must be fully documented in the case narrative notes.

5.2.3. Compound Quantification

Target compound concentrations are calculated using the following equations:

5.2.3.1. Aqueous samples

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

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

where:

A_x = Response for the analyte in the extract, in area or height units.
A = Amount of standard injected or purged in ng.
A_s = Response for the external standard, same units as A_x.
V_i = Volume of extract injected, µL. For low level purge and trap analysis, $V_i = 1$. For medium level purge and trap analysis there is a V_i term.
D = Dilution factor of extract. The final result of an algebraic multiplication of the ratio of all dilution final volumes to initial volumes. For example, if and extract was diluted 100 µL to 1000 µL and subsequently diluted an additional 100 µL to 1000 µL, the expression would be: (1000/10) * (1000/10) = 100 * 100 = 10,000.
If no dilution was made, D = 1.
V_t = Volume of total extract, µL. For low level purge and trap analysis $V_t = 1$. For medium level purge and trap analysis, there is a V_t term.
V_s = Initial sample size, ml.

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

5.2.3.4. The equation then becomes:

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

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

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

5.2.3.6. The equations for internal standard calculations are

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

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

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

and

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

A_S = Response of target analyte
A_{iS} = Response of internal standard
C_S = Amount of target analyte, in ng
C_{iS} = Amount of internal standard, in ng

5.2.3.7. Non-aqueous samples

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

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

where:

W = Weight of sample extracted or purged, grams.

TS = Total solids, percent.

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

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

Where W_S = sample size prepared, in gm

6. Reports

6.1. Data Packet Organization

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

6.2. Quality Control Reports

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

6.2.2. Add in any other specific stuff here.

6.3. Sample Result Reports

6.3.1. Data Qualifying Flags

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

Code	Definition
6.3.1.1.	The analyte of interest was not detected, to the reporting limit indicated.
6.3.1.2.	The analyte of interest was detected in the method blank associated with the sample, as well as in the sample itself. The B flag is applied without regard to the relative concentrations detected in the blank and sample.
6.3.1.3.	The analyte of interest was detected below the practical quantification limit. This value should be regarded as an estimate.

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

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

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

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

6.4. Control Chart(s)

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

7. References

Use the following format:

7.1. *Reference Title*, Author, Publication date

APPENDIX I

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

Standard Solution Concentrations, units

lowest ---> ICV/CCV ---> highest

Compound	STD1	STD2	STD3	STD4	STD5	MDL

Calibration Stock Solution, units

Compound	Conc

Surrogate Stock Solution, units

Compound	Conc

IBLK solution, units

Compound	Conc

APPENDIX II

Analysis Sequence

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

(fill in this table in more detail)

last	CCV standard

APPENDIX III

Data Packet Check List

I. QC SUMMARY

_____ Analysts 'Client' Comment (hard copy and floppy)
_____ Surrogate Recovery Summary Report
_____ Blank Spike Report
_____ MS/MSD Report
_____ Method Blank Summary

II. SAMPLE DATA:

_____ Organic Analysis Data Sheet
_____ Sample Confirmation Worksheet
_____ Chromatograms, primary column
_____ Chromatographic Report, primary column
_____ Chromatograms, secondary column
_____ Chromatographic Report, secondary column

III. STANDARD DATA:

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

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

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

APPENDIX III, continued

Method Detection Limit Standard:

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

Other Standards Used to Support Sample Data and Instrument Blanks

V. Raw QC Data:

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

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

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

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

APPENDIX III, continued

V. Bench Sheets

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

VI. Reject Data:

DO NOT COPY DO NOT PAGINATE

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

APPENDIX IV

Method Validation Criteria

Compound Criterion	Recommended Concentration	RSD

APPENDIX V

QC Summary Table

Laucks Testing Laboratories

Method _____ QA Requirements and Corrective Actions

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

APPENDIX VI

Method Flow Chart:

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

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1002

Title: **Document Tracking and Control**

Revision history:

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

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

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

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SOP UPDATE FORM 17

1. Introduction and Scope

1.1 Purpose

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

1.2 Document Types

1.2.1 Laucks recognizes two types of documents.

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

1.3 Scope

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

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

2. Operation Procedures

2.1 Initiation and Updating of Documents

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

2.1.2 The form should be filled out by either the individual responsible for the creation or revision, their Department Supervisor, or Division Manager. Creation or revision of documents may also be assigned by the Laboratory Director, Technical Director, or QA Officer to specific individuals. The form, however, must be approved and kept on file by the QA Department.

2.1.3 Copies of this form will be given to the responsible individual and the appropriate Division Manager. Originals will be kept on file in the QA Department. This insures that all responsible parties are informed of the initiation of the creation or revision process. This form should be filled out as soon as it is determined that the creation or revision of a document is necessary and a responsible party has been assigned. These forms will also be issued approximately annually in order to initiate the review process for existing SOPs.

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

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

2.2 Tracking and Control of Existing Documents

2.2.1 Most documents, particularly SOPs and administrative documents, will be assigned document numbers beginning with LTL. The scheme for numbering documents then proceeds as follows: The most important designator is the "thousands" place. If it is unclear which "hundreds" place designator is appropriate, the one which appears to be most appropriate may be used. This SOP will not be considered to have been violated if an incorrect "thousands" or "hundreds" place designator was used but every effort will be made to use the correct designators in order to maintain a more logical organization. This organization, although preferable, is not necessary for actual control of the documents as long as each complete LTL designator is unique. Unique numbering is enforced by the SOP database.

LTL-1000	QA / Administration
LTL-2000	Health and Safety
LTL-3000	Organic Extractions
LTL-4000	Sample Control,
-4100	Project Management
-4200	Document Management and Reporting
LTL-5000	Computer Systems (LIMS / MIS)
LTL-6000	Miscellaneous
LTL-7000	Metals Digestion
-7100	ICP Analyses
-7200	ICP/MS Analyses
-7300	Graphite Furnace Analyses
-7400	Flame Atomic Absorption Analyses
-7500	Cold Vapor Atomic Absorption Analyses
-7600	Gaseous Hydride Atomic Absorption Analyses
LTL-8000	Gas Chromatography, Volatiles
-8100	Gas Chromatography, Semivolatiles
-8200	GC / Mass Spectrometry
-8300	HPLC
-8400	Other Organic Analyses
LTL-9000	Conventional Chemistry- Titrimetric Analyses
-9100	Conventional Chemistry- Spectrophotometric / Instrumental Analyses
-9200	Conventional Chemistry- Gravimetric Analyses

2.2.2 Original documents will always be given a revision number of 0. Subsequent revisions, no matter how minor the revision, will be incremented by one.

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

2.2.4 SOPs, Methods, and many other documents must have header information which clearly indicates the document number, revision, date of revision, and document replaced by

revision. Page numbers are optional. The header may vary in format but must contain all required information similar to the following. SOPs which have been created using the template described in the Laucks SOP on the Elements of SOP and Method Formats will contain the header information as described.

SOP No: LTL-xxxx
Revision: 1
Date: 04/13/98
Page: x of xx
Replaces: 0

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

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

2.2.7 A database is maintained by the QA Department which, as a minimum, will track the document number, Department, revision number (or New or Draft if the document is incomplete), responsible individual, title and SOP Manual distribution (if the document has been completed and approved). Also tracked are the most recent revision date, the next revision due date, the last review date and, if any version existed under the previous SOP system, the previous SOP number. Only the applicable fields among these latter fields need be filled out. A copy of the screen form is presented in Appendix B.

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

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

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

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

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

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

- Newly distributed versions are placed in the SOP manuals.
- The signature lists for the current document are signed and dated as the staff complete reading the SOPs. In addition, as staff new to a particular task (SOP) are trained, the departmental supervisor will insure that they have read and signed the signature list for that SOP. This may require that the supervisor request a new SOP signature list for that staff member so that they can sign the SOP for newly assigned tasks.

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

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

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

2.3 Storage and Filing of Controlled Documents

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

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

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

2.3.4 Electronic versions of all controlled documents are also kept on file by QA. These versions are stored in an area of the laboratory network which has limited access to designated individuals. These electronic copies will be given names as closely matched as possible to their document or SOP number. Original documents and revisions will be given the extension .R0 or .R1, etc. to indicate their revision number. Should multiple files be necessary to create a given document, they will be incorporated into a subdirectory with similar naming conventions.

2.3.5 Copies of these electronic versions of SOPs will be distributed to individuals who have been assigned a revision. No other copies of these controlled documents should be kept by laboratory staff in order that unapproved copies of the document do not proliferate.

2.4 Review and Updating of Documents

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

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

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

Appendix A

Document Control Form

Laucks Testing Laboratories

DOCUMENT CONTROL FORM

_____ Generate new document

_____ Modify existing document

_____ Review existing document

Document No.: _____

Document Title: _____

_____

_____

Assigned to: _____ Date: _____

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

Reviewer: _____ Date: _____

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

_____

_____

_____

_____

_____

_____

QA Approval: _____ Date _____

Appendix B

SOP Database Screen

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

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

Current Document #: LTL-1002
Revision Date: 12/27/95
CHOOSE Department: Quality Assurance
Next Revision Due: 7/15/96
Revision #: 002
Last Review Date: 12/27/95
CHOOSE Supervisor Name: Romberg, Harry
Previous Document #: LTL-0054
Document Name: Document Tracking and Control.

Department Abbreviation: QA

Current SOP #: LTL-1002

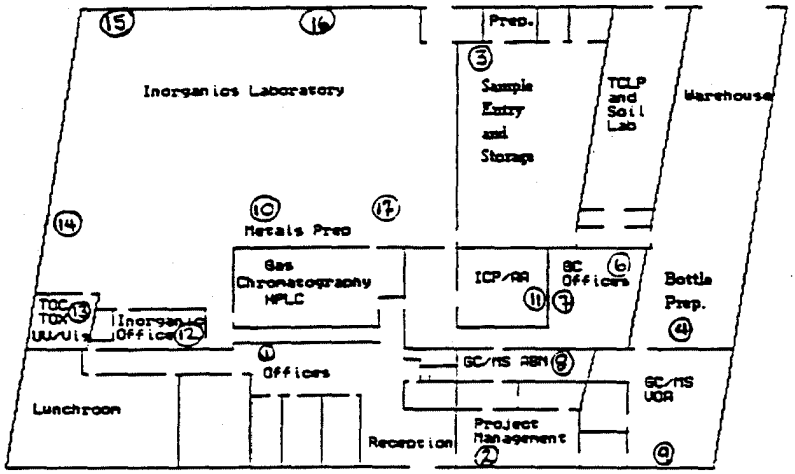
SOP Distribute: Yes

Record: 1 of 13

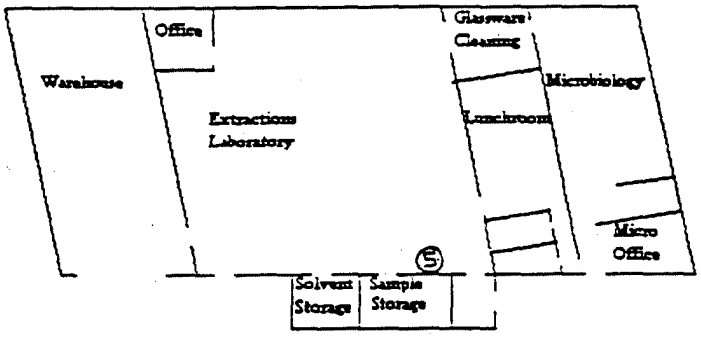
Appendix C

SOP Manual Distribution Locations

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



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



SOP Book Location

Appendix D

Document Control Stamp

Controlled Document

No. _____ Assigned to: _____

Appendix E

SOP Update Form

Laucks Testing Laboratories

SOP UPDATE FORM

Document No.: _____

Document Title: _____

_____

_____

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

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

Submitted by: _____ Date: _____

Approved by (QA): _____ Date: _____

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1003

Title: Chain-of-Custody and Documentation Procedures

Revision history:

Number	Date
1.	2/13/95
2	2/3/98

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

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

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APPENDIX A **8**

SECURE STORAGE CUSTODY LOG 8

APPENDIX B **10**

VOLATILES CUSTODY LOGS 10

1. Introduction and Scope

1.1 Description

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

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

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

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

1.2 Definition of Terms

1.2.1 Custody - A sample is considered under custody if:

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

1.2.2 Chain of Custody - The process by which custody of a sample is maintained and documented throughout the period that the sample is in the possession of the laboratory.

Any changes in the possession (custody) of the sample must be documented in order that the chain-of-custody can be properly maintained.

2. Equipment List

- Secure Storage Custody Log(s), see Appendix A
- Volatiles Custody Log(s), see Appendix B

3. Safety Precautions

3.1 Safety Precautions

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

4. Operation procedures

4.1 Identification of Samples Requiring Strict Chain-Of-Custody

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

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

4.2 Initiating Internal Chain-Of-Custody

4.2.1 Internal chain-of custody procedures begin when the samples are logged into the laboratory database. When the samples are logged into the system, they are stored in or near the sample entry area, in the main laboratory, in one of 3 locations:

- The main walk-in cooler is for organic extractables which have not yet been transferred to the extractions laboratory and for inorganics which require refrigeration.
- The volatiles refrigerators are located in the area between the GC room and the laboratory computer system hub.
- The locked "cage" in the log-in area is for samples not requiring refrigeration.

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

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

4.2.4 Samples requiring secure storage are logged into any of these areas by the sample receiving representative using a Secure Storage Custody Log (Appendix A). Samples not requiring secure storage need not have this form completed. A custody log will be completed for each **workorder** for which samples require chain-of-custody procedures.

4.3 Maintaining Internal Chain-Of-Custody

4.3.1 When samples are logged out of storage areas, they will be signed out in the appropriate spaces by the person removing them.

- If they are being removed for analysis, the "**Action**" column should state the analyses being performed. When they are returned, the logsheet must also indicate such. Additionally, the "**Sample Numbers**" column should indicate which samples are being removed for analysis (i.e. 1-10 metals digestion, or 3-5 NO3/NO2 analysis).
- If they are being removed for transferal to another location (i.e. extractions), the "**Action**" column should state where they are being transferred. Additionally, the "**Sample Numbers**" column should indicate which samples are being transferred (i.e. 1-10 volatiles, or 3-5 extractables).
- When samples are removed for final disposal, if all samples are being removed, the logsheet is signed and dated at the bottom of the page. If only certain samples are being disposed or to be even more clear, the "**Action**" column should indicate "disposed" and the "**Sample Numbers**" column should indicate which samples are being disposed.

4.3.2 When samples are signed into another storage location, this is done using an identical Secure Storage Custody Log. Samples which are subsequently removed from these areas for analysis or disposal should be signed out using the same procedures as above.

4.3.3 Volatiles samples, being generally for a single analysis, are signed out using an abbreviated logsheet. Copies of GC/MS and GC VOA logsheets are located in Appendix B.

4.3.4 Any analyst removing samples from **any** secure storage area for the purpose of preparation or analysis or transferal to another department **must** sign the samples out using the Secure Storage Custody Log and must sign the samples back in when they are returned, or must sign them into another secure storage area. Samples must be in the possession of the analyst who signed them out at all times during this period and must not be left unattended. If samples are analyzed and then immediately disposed, as may be the case for some volatiles analyses, the "**Action**" column on the custody log should indicate "analysis and disposal." Note, in checking volatiles samples out using a volatiles custody log, it is assumed that the purpose is to analyze for volatiles since no other analysis is performed on these samples.

4.3.5 There is ample room on any one Secure Storage Custody Log in almost all cases to accommodate checking all sample containers for a particular SDG or workorder in and out as often as required for all of the pertinent analyses from that secure area. Should an additional page be required, it should be obtained from Sample Entry. A second (or third) page must not be initiated until all of the space on the previous form has been filled. The first page must be marked "1 of 2" in the upper right corner and the second page marked "2 of 2". In the unlikely event that even more pages are required, this mark can be crossed out (single line, initialed and dated) and the sheets marked "1, 2, or 3 of 3, etc.

4.4 Sample Disposal and Closing of the Internal Chain-Of-Custody

4.4.1 When samples have been signed out for final disposal the chain-of-custody process is considered to be complete. The Secure Storage Custody Logs must be collated, bound and turned in to the Quality Assurance Department in order that the chain-of-custody can be tracked for all samples requiring this process, should such tracking be required at a later date.

4.5 Review of Custody Logs

4.5.1 On at least a quarterly basis, supervisory personnel must review selected custody logs to insure they are being filled out properly and completely. The supervisor need not review

all of the forms but must review at least 5% or 10 forms, whichever is greater. The supervisor will stamp the reviewed forms with a "reviewed by" stamp , initialing and dating the notation or may write "reviewed by" by hand, likewise initialing and dating the form. The supervisor should also cursorily review the remaining forms to make sure there are no blatantly obvious omissions but need not mark these forms unless a discrepancy is observed.

4.5.2 Review should include noting that all spaces are filled in properly and completely (this especially means that all samples that were checked out must be checked back in and that the person handling the samples must be identified), that errors are crossed out with a single line, initialed and dated, that entries are clear and not obliterated.

4.5.3 Should the supervisor find errors or omissions, the must issue a corrective action form to the individual who made the error (if it is obvious who that is) or to the appropriate supervisor. Any of these individuals can correct the form (initialing and dating the correction). The primary purpose of the corrective action notice is so that the individual in error be re-trained as to the proper custody procedure.

4.5.4 Supervisors responsible for this review may assign the responsibility to other capable individuals but the ultimate responsibility is theirs. These supervisors are the Organics Division Manager for the volatiles logsheets, the Extractions supervisor for the extractables, and the Sample Control supervisor for the main lab walk-in and unrefrigerated secure storage areas.

Appendix A

Secure Storage Custody Log

Laucks Testing Laboratories, Inc.

Secure Storage Custody Log

Project:_____ **LTL Number:**_____

Number of Containers (*optional*)**:**_____

Storage Unit:_____ **SDG Number** (*optional*)**:**_____

Sample Number	Matrix (*optional*)	Location (*shelf*)	Logged Out		By	Logged In		By	Action
			Date	Time		Date	Time		

Samples Disposed of by _____ on _____

Appendix B

Volatiles Custody Logs

LAUCKS TESTING LABORATORIES
GC/MS VOA CUSTODY LOG

LABORATORY NUMBER	SAMPLE NUMBER	DATE & TIME REMOVED	BY	DATE & TIME RETURNED	BY	COMMENTS

GC Volatiles Custody Log
Laucks Testing Laboratories, Seattle, Washington

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

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1004

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

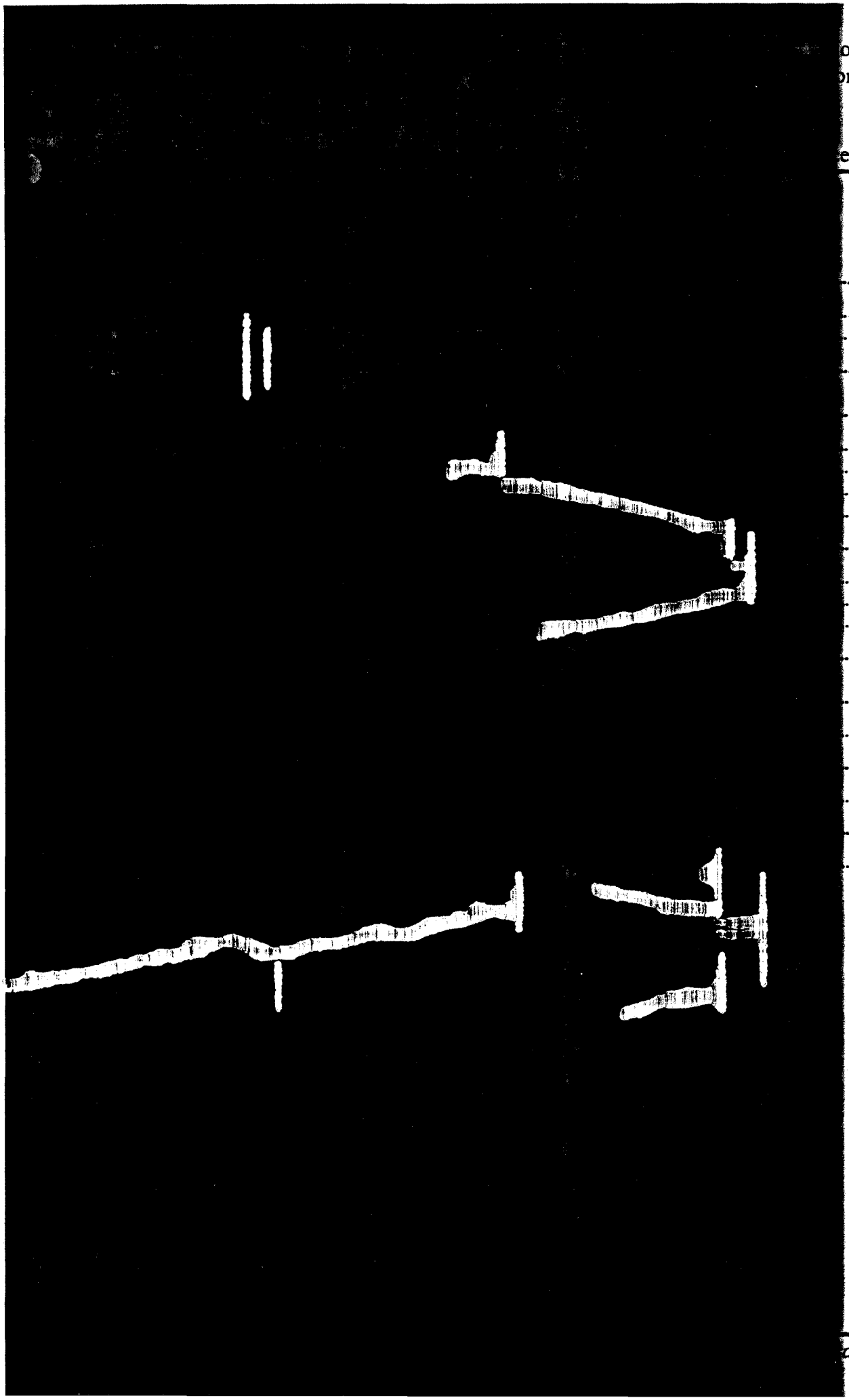
Revision history:

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

UNCONTROLLED



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1. Introduction and Scope

1.1 Description

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

1.2 *Scope*

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

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

2. Definitions

- ***PE** - Performance Evaluation*
- ***P&A** - Precision and Accuracy*
- ***Trainer** - An individual who has documentation demonstrating experience recognition or successful completion of competency and has been performing the task/method for a minimum of 3 months experience for login, sample preparation, and reporting and a minimum of 6 months for analytical instrumentation operation and analysis reporting.*

3. *Responsibilities*

3.1 *Analyst*

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

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

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

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

3.2 *Supervisor*

3.2.1 *It is the supervisor's responsibility to ensure that their analysts are all initially qualified to perform an analysis including ensuring that they have analyzed all required PE samples and performed all required P&A studies for the methods for which they will be doing analyses.*

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

3.2.3 *It is the supervisor's s responsibility to ensure that on a continuing basis, at least annually, that analysts who are to be considered capable of performing an analysis, have performed within limits on at least one PE study for analyses for which such are available.*

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

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

3.2.6 *It is the supervisor's responsibility to ensure that the analyst's training file has been updated with the most current PE or P&A data as well as any quizzes or checklists that are considered part of their departmental training.*

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

3.3 *QA*

3.3.1 *QA maintains training files (except for Extractions where the supervisor maintains the files due to the location of the extractions facility).*

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

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

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

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

3.4 *Trainer*

3.4.1 *Completes applicable staff training documents during the training process.*

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

4. Operation procedures

4.1 Recognition of Experience and Training

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

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

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

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

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

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

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

4.2 Demonstration of Capability to Perform Analysis

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

4.3 *Ongoing Demonstration of Performance*

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

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

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

5. *References*

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

Laucks SOP

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

Appendix A

Recognition of Experience and Training Form

Recognition of Experience & Training Form

Laucks Testing Laboratories

It is hereby recognized that _____

Employee Name

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

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

_____ Division Manager

_____ Date

Appendix B

Demonstration of Capability to Perform Analysis Form

Demonstration of Capability to Perform Analysis

Laucks Testing Laboratories

Analyst:_____

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

Method:_____ PE Material:_____

Target Value:_____ Recovery Criteria:_____

Reproducibility Criterion:_____

Date	Result	% Recovery
%RSD:		

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

☐ Met Criteria ☐ Did Not Meet Criteria

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

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

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

_____ _____
Division Manager Date

Appendix C

Example Training Checklists

Laucks Testing Labs
Pesticide/Herbicide GC Semivolatile Analyst Training Verification Checklist

	Analyst Name:	Date:	Trainer:	Supervisor:	Analyst:
	umentation				
	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				
	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

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				
	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 check system flows				
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 clean P&T and autosampler lines*				
16	NON-ROUTINE: *Able to change nickel tubing, resin and IPA*				
Analytical Performance					
17	Able to prepare standards & pass standard QC acceptance criteria				
18	Able to analyze and generate acceptable calibration curve				
19	Able to analyze CCVs and apply QC acceptance criteria				
20	Applies acceptance criteria for surrogates and spikes				
21	Able to set up analytical runs & acquire data				
22	Able to get information on samples/analyses (test codes, MDLs, etc.)				
23	Able to quantitate an analytical batch (standards, CCVs, QC & samples)				
24	Knows how to confirm detection of analytes (peak ID, conf. col.)				
25	Knows reanalysis 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 volatile 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
Fuels GC Semivolatile Analyst Training Verification Checklist

	Analyst Name:	Date:	Trainer:	Supervisor:	Analyst:
	Documentation				
	Able to use Standards Log				
2	Able to use Instrument Run Logs				
3	Able to use Instrument Maintenance Logs				
	Methods				
4	Has read and understands SOPs for all applicable methods				
	List 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				
	Able to prepare standards & pass standard QC acceptance criteria				
	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.

Laucks Testing Labs
HPLC Semivolatile Analyst Training Verification Checklist

Analyst Name:		Date:	Trainer:	Supervisor:	Analyst:
Documentation					
1	Able to use Standards Log				
2	Able to use Instrument Run Logs				
3	Able to use Instrument Maintenance Logs				
Methods					
4	Has read and understands SOPs for all applicable methods				
	List Method(s):				
5	Has read and understands EPA Methods (SW846)				
	List Method(s):				
6	Has read and understands appropriate sections of HPLC Training Manual				
Instrument Operation/Maintenance					
7	Knows location and use of Instrument Manuals				
8	Knows basic HPLC theory				
9	Able to use solvent delivery system to set mobile phase program				
10	Able to use Autosampler Control Pad to set injection program				
11	Able to change filters and guard column				
12	Able to change columns & perform leak checks				
13	Able to measure and set mobile phase flows				
14	Able to prime pumps				
15	Able to prepare mobile phase (filter water, select correct solvent grade)				
16	Able to change Helium tank				
17	NON-ROUTINE: *Able to change lamps*				
18	NON-ROUTINE: *Able to locate the high pressure build-up*				
19	NON-ROUTINE: *Able to change pump seal*				
20	NON-ROUTINE: *Able to clean flow cell*				
Analytical Performance					
21	Able to prepare standards & pass standard QC acceptance criteria				
22	Able to analyze and generate acceptable calibration curve				
23	Able to analyze CCVs and apply QC acceptance criteria				
24	Applies acceptance criteria for surrogates and spikes				
25	Able to set up analytical runs & acquire data				
26	Able to get information on samples/analyses (test codes, MDLs, etc.)				
27	Able to quantitate an analytical batch (standards, CCVs, QC & samples)				
28	Knows how to confirm detection of analytes (peak ID, conf. col.)				
29	Knows reanalysis and reextraction criteria				
30	Able to perform sample dilutions (obtaining linear results)				
31	Knows correct reporting limits for method(s)				
32	Knows corrective action & documentation for out of control QC events				
33	Able to produce a data package (In-house and SW-846)				
Method Validation *(complete one or more of the following)*					
34	Has successfully analyzed four P&A samples				
35	Has successfully analyzed two PE samples				
36	Has successfully analyzed three each of two types of QC samples				

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

_____

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

Laucks Testing Labs

Semivolatile Analyst Training Verification Checklist

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			
	Able to check spectra vs. standard spectra			
17	Applies acceptance criteria for surrogates, spikes, & ISs			
18	Knows the basic differences between In-House, CLP, and SW-846			
19	Able to generate basic CHRO forms packages			
20	Knows where to get information on samples (test codes, etc.)			
21	Able to calculate RFs and results from raw data			
22	Knows the types of extraction procedures used for ABNs			
23	Knows basic GC/MS theory			
24	Has read and understands the SOPs for all applicable methods			
25	Knows corrective action for out of control QC events			
26				
27				
28				
29				

Work Order:	TICs?	Package SDG.	TICs?

GC/MS Semivolatile Analyst Competency Criteria

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

GC/MS Training Program

Criteria for Demonstration of Analytical Competency

Analyst Name: _____

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

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

Completed on : _____ Supervisor: _____

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

Completed on : _____ Supervisor: _____

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

MS/MSD _____
SRM _____
Blank Spike _____

Completed on : _____ Supervisor: _____

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

REPORT MANAGEMENT

Employee Name: Date of Hire:

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

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1005

Title: **Analytical Balances**

Revision history:

Number	Date
LTL-0005 R1	09/25/86
LTL-0005 R2	11/3/87
3	06/22/96
4	5/15/98

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

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

Controlled Document

No. 20 Assigned to: Tetra

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1. Introduction and Scope

1.1 General

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

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

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

2. Equipment List

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

3. Safety Precautions

3.1 Safety

3.1.1 So as not to expose themselves or other analysts to potential harm and in order not to cross-contaminate samples. **it is critical that the individual analyst clean the balance and the balance area after each and every use of the balance**.

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

3.1.3 Weighing chemicals and samples is potentially hazardous. The analyst should take every precaution to avoid contact of any of these things with the skin, eyes, or through

inhalation. In addition, the analyst should take precautions to see that nearby analysts or those using the balance afterwards are not inadvertently exposed.

4. Operation Procedure

4.1 Balance Setup

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

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

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

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

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

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

4.1.7 All doors to the weighing compartment should be closed.

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

4.2 Balance and Weight Calibration

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

4.2.1.1 **Daily** - The daily calibration is done by the **first user** of the day. The user places a tare weight on the balance equivalent to a tare typically used on that balance, weighs the daily standard (a class "S" weight typical of the weight used on that balance) and

records the weight in the balance record book. If the weight is outside the limits set for the standard, it must be brought to the attention of the area supervisor and QA.

4.2.1.2 **Weekly** - The balance will be checked with a range of class S weights each week by the laboratory balance custodian. If a reading for a given weight exceeds the limits for that weight, the balance custodian will bring it to the attention of the area supervisor and QA.

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

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

4.2.2.1 The Class "S" Weights - These are the primary standards for checking the accuracy of the balance. They must be handled with care as they are calibrated and damage to the weights may result in inaccurate balance calibration. These weights must only be touched with the forceps supplied with the weights or with the clean white gloves also kept with the weights. The class "S" weights are sent annually to a qualified weight re-certification service, currently Denver Instruments, although another qualified service is allowable. During this time the calibrations will be suspended or other Class "S" weights used (if available) until the calibrated weights return.

4.3 Responsibilities

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

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

- The user will report any malfunction or failure of the daily check to the area supervisor.
- The user will mark and not use any balance which has failed calibration.

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

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

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

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

4.4 Daily Calibration Check

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

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

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

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

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

4.4.6 A standard weight of a size commonly used on that balance must then be added and the weight relative to the tare recorded under the appropriate day of the week in the calibration logbook. He or she will also initial and date the entry (See Appendix I). The standard weight will be a class "S" weight or equivalent.

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

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

4.4.7.1 **Example 1:** 1 gram samples are typically weighed into flasks with tare weights of 100 grams on a balance weighing to 0.0001 g. In order to perform the daily calibration check, a flask of about 100 grams is placed on the balance and the weight recorded. The balance is tared (set to zero) based on this weight. A 1.0000 gm. Class "S" weight is then placed on the balance with the flask and the weight recorded. This second weight must read within the limits of 0.9995 gm to 1.0005 gm.

4.4.7.2 **Example 2:** 30 gram samples are typically weighed into beakers with tare weights of 80 grams on a balance capable of weighing to 0.01 grams. In order to perform the daily calibration check, a beaker weighing about 80 grams is placed on the balance and the weight recorded. The balance is tared (set to zero) based on this weight. A 30.0000 gm. Class "S" weight is then placed on the balance with the flask and the weight recorded. This second weight must read within the limits of 29.98 gm to 30.02 gm.

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

4.4.9 An example logbook page is presented in Appendix I

4.5 Weekly Calibration Check

4.5.1 The balance custodian is the person responsible for performing the weekly calibration check and reporting problems to the area supervisor or QA. The custodian may be a different person in each area and it is the responsibility of the area supervisor to ensure that a capable balance custodian has been assigned to each area for which they

responsible. It is the responsibility of the custodian to insure that the weekly check is done even if they are not present, such as for vacation, etc.

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

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

4.5.4 The balance custodian will insure the balance is clean.

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

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

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

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

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

4.5.9 An example logbook page is presented in Appendix I

4.6 Annual Calibration Check

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

5. References

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

APPENDIX I

Sample Page from a Balance Logbook

	TV	Class "S" Read		Mon.	Tues.	Wed.	Thurs.	Fri
May 4 - May 9, 1998	2.0	2.00	Tare →	54.17	54.16	54.17	54.18	54.15
	10.0	10.00	2.0 g →	2.00	2.00	2.00	2.00	2.00
DHR	50.0	50.00		DHR	DHR	DHR	DHR	DHR
5-4-98	150.0	150.00		5-4-98	5-5-98	5-6-98	5-7-98	5-8-98
May 11 - May 16, 1998	2.0	2.00	Tare →	54.17				
	10.0	10.00	2.0 g →	2.00				
DHR	50.0	50.00		DHR				
5-11-98	150.0	150.00		5-11-98				

Done once per week

Initial + Date required

Row Heading

Daily Weights

Initial + Date required

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1006

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

Revision history:

Number	Date
1	05/01/96
2	3/20/98

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

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

Controlled Document
No. 20 Assigned to: Tetra

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EXAMPLE COLD STORAGE TEMPERATURE LOG 13

1. Introduction and Scope

1.1 Method Description

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

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

2. Equipment List

2.1 Equipment

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

3. Safety precautions and Waste Disposal

3.1 Safety Precautions

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

3.2 Waste Disposal

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

4. Thermometer Purchasing and Identification

4.1 Purchasing

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

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

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

4.2 Identification

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

5. Calibration

5.1 Recalibration of the standard thermometer.

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

5.1.2 Note: Microbiology NIST traceable thermometers are recalibrated at the frequency required by the Washington State Department of Health, every 3 years.

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

5.2 Recalibration of Cold Storage and Room Temperature Thermometers

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

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

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

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

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

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

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

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

5.2.8 The differences in temperature between the standard thermometer and the individual thermometer are calculated and recorded in the log as the "correction factor". The correction factor is calculated as the standard thermometer reading minus the individual thermometer reading so that by adding the resulting number to the individual thermometer reading will result in a "correct" temperature.

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

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

5.3 Recalibration of Oven and Other Thermometers

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

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

5.3.3 The differences in temperature between the standard thermometer and the individual thermometer are calculated and recorded in the log as the "correction factor". The correction factor is calculated as the standard thermometer reading minus the individual thermometer reading so that by adding the resulting number to the individual thermometer reading will result in a "correct" temperature.

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

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

5.4 Recalibration of the Infrared Thermometer

5.4.1 The standard thermometer is placed in a glass Erlenmeyer flask filled with water in a cold storage unit at least overnight (12 hours).

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

5.4.3 The emissivity of the IR thermometer is set at the level determined in the previous calibration (if any). It should read the same temperature as the standard thermometer. If it doesn't, the emissivity is adjusted until the standard thermometer and the IR thermometer agree as closely as possible.

5.4.4 This emissivity setting, the calibration date and the person who performed the calibration are recorded on a label which is attached to the thermometer. Analysts subsequently using the thermometer must measure against a glass container and use the emissivity setting noted on the IR thermometer in order to get an accurate temperature measurement.

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

6. Monitoring Responsibilities

6.1 Use Of Calibration Logs For Cold Storage Monitoring

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

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

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

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

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

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

6.2 Monitoring Ovens And Other Devices

6.2.1 When oven thermometers are calibrated, QA will mark the oven with the thermometer ID and the correction factor. Log sheets are generally not used for ovens. It is the responsibility of any analyst using an oven to apply the correction factor when recording temperatures on data sheets.

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

7. Specification Limits and Corrective Actions

7.1 Thermometer Criteria

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

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

7.2 Thermometer Corrective Actions

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

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

7.3 Cold Storage Criteria

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

7.4 Cold Storage Corrective Actions

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

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

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

7.4.4 If the above fail to correct the problem, contact the laboratory maintenance personnel, the departmental supervisor or QA to arrange for repair.

7.4.5 If it is determined than professional servicing is required this may be arranged upon direction of one of these individuals or another senior supervisor. If professional maintenance does not correct the problem, the unit may need to be replaced, again at management discretion.

7.4.6 Samples must not be stored at inappropriate temperatures. If the problem is not quickly solved, samples or standards must be transferred to another cold storage unit. If it is determined that samples were stored at inappropriate temperatures for an extended period, it may be necessary to contact clients to determine the course of action they would like us to take regarding their analyses. This should be coordinated with QA and project management. Standards which have been inappropriately stored will generally require disposal, generally at the discretion of QA and/or department managers.

7.4.7 Any corrective actions (including simple adjustments) **must** be noted on the Cold Storage Temperature Log (Appendix B).

Appendix I

Example Calibration Spreadsheet

Laucks Testing Laboratories
1997 Laboratory Thermometer Calibration

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

Laucks Testing Laboratories
1997 Laboratory Thermometer Calibration

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

Appendix II

Example Cold Storage Temperature Log

Cold Storage Temperature Log

Laucks Testing Laboratories, Inc.

Cold Storage ID #:_____ Thermometer ID:_____

Location:_____ Year: **1998** Correction Factor (*add this number when* ***recording*** *the thermometer reading):*_____°C

Day	Month: Time	Temp.	Initials	Actions	Month: Time	Temp.	Initials	Actions	Month: Time	Temp.	Initials	Actions
1												
2												
3												
4												
5												
6												
7												
8												
9												
10												
11												
12												
13												
14												
15												
16												
17												
18												
19												
20												
21												
22												
23												
24												
25												
26												
27												
28												
29												
30												
31												

Record Time and Temperature in the proper blocks and initial the entry each day of normal laboratory operation. Q:\TEMPLATS\LTLFORMS\REFERFRM.DOC. 3/19/98

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

Corrective action includes 1) Adjust the temperature of the thermostat 2) Defrost the refrigerator or freezer

3) Contact the appropriate laboratory maintenance personnel, the departmental supervisor, and/or the QA Officer

4) One of the above may decide that professional maintenance is necessary or even that the cold storage unit must be disposed of.

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

Samples **MUST NOT** be stored in units which are not maintaining the proper temperature.

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1007

Title: Use of Instrument Records and Logbooks

Revision history:

Number	Date
1.0	9/5/91
2	5/13/98

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

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

Controlled Document

No. 20 **Assigned to:** Tetra

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1. Introduction and Scope

1.1 Introduction

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

1.2 Scope

1.2.1 This SOP primarily addresses instrument run log maintenance, maintenance manuals and other logs not addressed in other SOPs. Standards log, for instance, are discussed in the standards SOP, LTL-1013. Analytical balance logs are discussed in that SOP, LTL-1005. Cold storage logs are discussed in LTL-1006. Control and monitoring of logbooks and general items pertinent to all logbooks is discussed in Laucks SOP LTL-1019.

1.3 Definition of Terms

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

2. Equipment List and Standards

2.1 Equipment

2.1.1 maintenance logbook, analytical run logbook (where appropriate) or other applicable logbook

2.1.2 pen (pencil is NOT allowed)

3. Operation procedures

3.1 All Logbooks

3.1.1 All logbooks should be numbered and controlled according to procedures outlined in Laucks SOP LTL-1019. It is the analysts responsibility before initiating any new

logbook to ensure that the logbook has been identified and given a logbook number by QA. See LTL-1019 for further detail.

3.1.2 **NOTE:** All errors in all logbooks must be altered by a single-line crossout which must also be initialed and dated. No erasures, overwriting, white-out or multiple-line crossouts (blacking out) are acceptable.

3.1.3 **NOTE:** Empty space in logbooks must be lined out (preferably with a Z for large blocks of empty space). This mark, as with error correction, should be initialed and dated.

3.2 Maintenance Manuals

3.2.1 All instruments at Laucks from GC or GC/MS systems to ICPs, AAs, spectrophotometers, ion chromatographs, etc. have instrument maintenance manuals associated with the specific instrument.

3.2.2 Maintenance manuals are bound notebooks with the specific instrument and, if appropriate where multiple similar instruments are involved, instrument names or numbers printed on the outside cover. If there are multiple books for an instrument, which may be the case for instruments which have been in service for a long time, especially if they have required extensive, ongoing maintenance, the notebooks should be clearly numbered on the cover as #1, #2, etc.

3.2.3 As a general rule, loose leaf or 3-ring bound notebooks are not acceptable. The exception to this rule is for maintaining copies of professional service call paperwork or if specific forms have been created for monitoring maintenance activities. Such paperwork must be dated. Note of the service should still be made in the bound notebook associated with that instrument and the identifying number on the service log noted in the maintenance manual.

3.2.4 With a few basic rules, these maintenance manuals are free-form with no specific format but MUST include any and all maintenance associated with the particular instrument.

3.2.4.1 Each entry should be INITIALED by the person making the entry.

3.2.4.2 The maintenance manual must contain the DATE any service or maintenance was performed on the instrument and exactly WHAT that operation was. This includes everything from changing a part to cleaning an instrument orifice or changing a chromatographic column or instrument tubing. It should include everything from the simplest maintenance to the most complex, including any professional service calls.

3.2.4.3 Where maintenance is routine, some books use codes for the most common service operations. These codes must be clearly defined either on the front, inside cover of the maintenance manual or on the first page. If there are multiple books, these codes must be so defined in EACH book.

3.2.5 If the maintenance was performed because of a specific problem (not just routine, ongoing maintenance) the problem should be described in at least one entry in the maintenance book as well as the work performed at any one time, and the outcome of that maintenance, that is whether or not it was successful or what occurred when the work was performed.

3.2.6 In order to aid in monitoring instrument performance changes, service or equipment changes may also be noted in instrument run logs. However, this information is supplementary. ALL maintenance must be recorded in the maintenance manual.

3.3 Instrument Run-Logs

3.3.1 Instrument run-logs come in two essentially different forms, with variations depending upon the specific instrument. In any form, a copy of the daily run log must accompany the data from each laboratory workorder for any samples associated with that sequence.

3.3.2 GC, GC/MS, HPLC, GPC and other run-logs are in bound, pre-printed, sequentially page-numbered books. They are identified by the specific instrument type and, if appropriate where multiple similar instruments are involved, instrument names or numbers printed on the outside cover. If there are multiple books for an instrument, which will be the case for instruments which have been in service for very long, the notebooks should be clearly numbered on the cover as #1, #2, etc.

3.3.2.1 Run logs must identify the method being run either at the top of the page, or if more than one method is being used for any sequence, clearly marked by the sample entry. It is recognized that it is in some cases possible to use different methods, which may only be different in the way a calibration is interpreted or validated. It may even be that two methods are essentially identical. However, in these instances, the logsheet should clearly indicate for which method a particular sample is being analyzed.

3.3.2.2 Instrument run-logs should include places to record all relevant sample and data file IDs, performance criteria, sample type and size, additional comments pertinent to the specific analyses, and analyst initials. All appropriate information must be filled out and the page dated. Examples of current logbook forms (at the time of this writing) are located in Appendices I (GC/MS), II (GC and HPLC), and III (GPC). These forms should be considered examples and not as the only forms used by Laucks for this purpose. These forms may change with approval of the department manager and QA. Although this SOP

will not then be considered invalid, new example forms should be incorporated into the next revision.

3.3.3 In addition to the appropriate header information for each analytical GC, GC/MS, HPLC, GPC or other run, all of the pertinent information should be filled out for each injection.

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

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

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

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

3.3.8 A copy of the run-log is included with each data packet associated with that run.

3.3.9 As with the bound book format, the samples, standards, calibration checks, reference materials, etc. should be identified and listed IN ORDER.

3.3.10 Information critical to identifying the analytical run (date, analyst, analysis type) must be included in the header information. If multiple analytical runs were made in one day, they must be identified as run #1, run #2. etc. If the instrument is capable of time-stamping run data, this option should be utilized, although it need not be included in the run-log itself.

3.3.11 Where possible laboratory practice is to maintain ongoing run-logs for inorganic instrumentation. The daily run-logs are included with all data. Records which do not lend themselves to being kept in a pre-printed bound logbook may be collected in a 3-ring binder in an organized format but not unbound or loose-leaf. After sufficient logs have been collected, they should be bound with the laboratory comb binder. These logs should be given QA logbook IDs as described in Laucks SOP LTL-1019.

3.4 Other Logbooks

3.4.1 The same general principals used for the above logbooks apply to any other logbook, unless otherwise defined in a specific SOP.

3.4.1.1 Entries should be initialed and dated.

3.4.1.2 Empty space between entries should be minimized

3.4.1.3 Errors and empty spaces should be properly crossed out, initialed and dated.

3.4.1.4 Pages are preferably sequentially numbered but if this is not practical, at least dated and/or time stamped.

3.4.1.5 The logbook should identify the operation being monitored.

3.4.1.6 The pages in the logbook should contain all appropriate information needed to identify the activity and all applicable spaces should be completely filled out.

3.4.1.7 The logbook should be given a QA ID number as described in LTL-1019.

Appendix I

GC/MS Run Logs

Laucks ... ng Labs, Inc.
GC/MS VOA ... trument Run Log

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

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

624 8260 524 SIM CLP-Low CLP

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

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

Laucks Testing Laboratories

GC/MS Semivolatiles Injection Logbook

IS Std ID_____

CCV ID_____

DFTPP ID_____

Page: 5699

Date:_____

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

Appendix II

GC/HPLC Run Logs

Laucks Testing Labs

GC VOA Instrument Log

Analyst : _____ Date : _____

Chromatography Ref. Page : _____ Calibration Reference : _____

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

LAUCKS TESTING LABORATORIES

INSTRUMENT LOGSHEET

Analyst : _____ Date : _____

Instrument ID : _____ Column 1: _____

Injection Volume : _____ ul Column 2: _____

Calibration Standard Reference : _____

File ID	Sample ID	DF	Comments

LAUCKS TESTING LABORATORIES

HPLC INSTRUMENT LOGSHEET

Analyst : _____ Date : _____

Instrument ID : _____ Column : _____

Injection Volume : _____ ul Solvent : _____

Calibration Standard Reference : _____

File ID	Sample ID	DF	Matrix	Comments

Appendix III

GPC Run Log

Laucks Testing Labs, Inc.
GPC Bench Sheet

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

Operating Conditions:

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

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

Comments:

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1008

Title: **QC Corrective Action**

Revision history:

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

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

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

UNCONTROLLED

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EXAMPLE LOGSHEET (COLD STORAGE) 14

1. Introduction and Scope

1.1 Purpose

1.1.1 The purpose of this SOP is to establish a system to identify, document and resolve out-of-control events.

1.2 Scope

1.2.1 An out-of-control event may be recognized by any member of Laucks. When they occur, the analyst, supervisor and Quality Assurance work jointly to solve and correct the problem. Out-of-control events are documented using an Out-of-Control-Event form or a Corrective Action form, or in a few selected instances, on a logsheet with space specifically for such actions. Corrective action resulting from an audit is also dealt with using its own Audit Response form but this action is elucidated in an SOP specific to that process.

2. Definition of Terms

2.1 This section defines terms and acronyms as they are used in this SOP.

2.1.1 **Corrective Action**: Action taken by an individual(s) to correct a problem as evidenced by either the failure of QC criteria or a more general problem which could affect performance of an analysis, the quality of service or other activity undertaken by the laboratory.

2.1.2 **Out-of-control event**: Any occurrence or condition failing to meet Laucks QC criteria or has the potential to impact data quality.

2.1.3 **QA/QC**: Quality Assurance/Quality Control

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

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

2.1.6 **RPD**: Relative Percent Difference

2.1.7 **LCS**: Laboratory Control Sample

3. OUT-OF-CONTROL EVENT PROCEDURE

3.1 Identifying an Out-Of-Control Event

3.1.1 The following is a list of examples of out-of-control events. This is not a complete list of all possible out-of-control events and many of those listed may be different for different methods. Specific criteria are given in analytical SOPs or in other QA documents. If there is doubt about whether a situation is out-of-control and must be responded to, consult with Quality Assurance.

3.1.1.1 GC/MS instrument tune criteria failing to meet criteria

3.1.1.2 Initial calibration linearity, depending upon the method used for calibration, correlation coefficient <0.995 (<0.990 for some fuels analyses) or percent RSD failing to meet method specifications.

3.1.1.3 Daily and continuing calibration verification or calibration blanks outside acceptable ranges as defined in their respective SOPs.

3.1.1.4 **NOTE:** If any of the above instances (3.1.1.1-3.1.1.3) occurs, analysis is stopped. No sample analysis can occur until the event is back in control. A corrective action form does not need to be filled out for these instances if identified at the analyst level and corrected before any data are affected.

3.1.1.5 Matrix spike, surrogate spike or blank spike recoveries outside acceptable ranges.

3.1.1.6 Unacceptable RPD value for MS/MSD or duplicate samples.

3.1.1.7 Unacceptable values for LCS's and QC samples.

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

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

3.1.1.10 **Note:** Samples which contain target analyte levels which are greater than 20 times the blank or which contain none of the offending analyte may be considered acceptable.

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

3.1.1.12 A sample depleted before all required analyses are completed.

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

3.1.1.14 Contaminated reagents and glassware.

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

3.1.1.16 Record keeping omissions, errors, and deviations from the record keeping standard operating procedures are also out-of-control situations

3.2 Responding to an Out-Of-Control Event

3.2.1 When an out-of-control event is recognized, each individual involved with the analysis in question has an interactive role and responsibility, these are as follows:

3.2.2 Analyst:

3.2.2.1 Must be able to recognize QC failure and immediately take the proper action or, if unsure of the appropriate response, notify the supervisor and work with the supervisor and Quality Assurance to solve the problem; also maintains QC charts.

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

3.2.2.3 Examine all calculations for correctness

3.2.2.4 Examine bench sheets for correctness

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

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

3.2.2.7 Re-analyze the sample

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

3.2.2.9 If these steps do not yield acceptable results, consult the supervisor.

3.2.3 Supervisor:

3.2.3.1 Must review all analytical and QC data for reasonableness, accuracy and clerical errors; also responsible for QC charts. Some of the above duties may be assigned to others, with supervisory oversight, if those others have been trained to observe the conditions which would initiate further investigation.

3.2.3.2 In an out-of-control event, the supervisor works with the analyst and Quality Assurance to solve the problem and prevents the reporting of suspect data by stopping work on the analysis in question and insuring that all results that are suspect are repeated, if possible, after the source of the error is determined and remedied.

3.2.3.3 If corrective actions do not yield results which meet specifications, it may be determined that sufficient action has been taken. The supervisor and QA will approve of such decisions and if it is determined that the data quality could be impacted, the supervisor will ensure that appropriate comments are reported with the data to the client.

3.2.4 Quality Assurance:

3.2.4.1 The Quality Assurance Officer or designee will work with supervisory personnel and/or analysts to solve out-of-control situations which are not routinely corrected at the bench.

3.2.4.2 In the event that an out-of-control situation occurs that is unnoticed at the bench or supervisory level, such as performance failure on a blind QC sample, Quality Assurance will notify the supervisor, help identify and solve the problem where applicable, insure the work is stopped on the analysis and no suspect data is reported.

3.2.4.3 Finally the Quality Assurance Officer or designee must review and approve all corrective action reports which cannot be resolved. If corrective actions do not yield results which meet specifications, it may be determined that sufficient action has been taken. The supervisor and QA will approve of such decisions.

3.2.4.4 If it is determined that the data quality could be impacted, the supervisor will ensure that appropriate comments are reported with the data to the client and QA will review said comments.

3.2.5 Project Manager:

3.2.5.1 The Project Manager is responsible for notifying the client of out-of-control events, such as missed holding times, raised reporting limits, matrix interferences, etc. which cannot be resolved without potential impact on either the data quality, the agreed upon or routinely reported results, or the timely and expected delivery date. It is not necessary to contact the client for events which are correctable and do not impact the final data quality, holding times or turn-around unless specifically requested by the client.

3.3 Corrective Actions

3.3.1 Appropriate corrective action depends on the type of analysis, the extent of the discrepancy, and whether the event is determinant or not. The corrective action to be taken for analytical QC failures is usually described in the specific analytical method but may also be determined by either the supervisor, Quality Assurance Officer, or by both in conference, if necessary.

3.3.1.1 Some items may not necessitate direct intervention of QA where standard practices are in place for some events, where the SOP or project or program QAP itself dictates the corrective action and where the action taken is the most conservative response practical. These types of events may be considered to have automatic QA approval and may not even require the completion of any related out-of-control event forms.

3.3.2 A corrective action can be as extensive as replacing a complete lot of contaminated extraction solvent, re-extracting and re-analyzing a complete batch of samples, due to reagent blank contamination; or as simple as recalculating a series of results because a wrong dilution factor was applied. Again, the appropriate corrective action must be determined on a case by case basis.

3.3.3 Data cannot be released until the system is in control or the QC failure can be attributed to a cause other than method performance. In the event the out-of-control event is due to matrix problems in the sample, and the system remained out of control, the data is flagged and supporting documentation is released to the client.

3.3.4 Corrective actions are considered adequate when the problem has been resolved and data can be reported or other actions taken from an in-control condition. Alternatively, it may be determined that the action taken was, as a minimum, all that was required by the method or that no further action was reasonable or possible that would improve the data. In these cases, the final decision must be approved by the supervisor and QA.

3.4 Documenting an Out-Of-Control Event

3.4.1 This is accomplished by completing one of the following

- A Corrective Action (CA) Form (See Appendix 1)
- A QC_DB Report Form (for Inorganics analytical QC only, see Appendix 2)
- An Out-Of-Control Event (OOCE) Form (lab use only, see Appendix 3)
- A Sample Receipt Form (for sample receipt events, see Appendix 4)
- An Audit Finding Report Form (QA use only, not shown here, see audit SOP)
- or logged onto a form which itself includes corrective actions (example, Cold Storage Logsheet, see Appendix 5).

3.4.2 CA forms are general and are for documenting corrective action taken to correct problems not associated with a particular analytical event.

3.4.3 Out-Of-Control Event (OOCE) Forms are filled out by **technical** laboratory staff only and are designed for documenting analytical QC failures and associated corrective actions. Where other forms, such as the Inorganics QC_DB Report Form, are used to document that the QC parameters were checked, any failures of QC and the decision to perform corrective action or continue data processing must be documented on the OOCE form. The checklist may then be attached to the OOCE form for final data submission.

Note: It is not necessary for analytical staff to document actions which were taken prior to processing samples or which do not affect reported data.

3.4.4 Audit Finding Reports are responded to by the assigned individual and signed off by QA or a designated individual (see the audit SOP).

3.4.5 All OOCE and Corrective Action Forms shall be filled in completely by the person observing the event. Actions taken may be filled in by either the initiating person or the person actually performing the corrective action. The descriptions of the event and any corrective actions taken should be detailed and specific. The OOCE form provides check boxes for most analytical events.

Note: Holding time violations due to laboratory error are annotated on the OOCE form. Holding time violations occurring due to receipt of samples beyond the criteria are documented on the sample receipt form only.

3.4.6 If the corrective action taken and annotated on the OOCE Form resolves the problem and allows data to be reported which is in control, the action is complete and only needs to be signed by the individual taking action and the individual initiating the action.

3.4.7 If the corrective action taken and annotated on the OOCE Form does not resolve the event and it is determined that no further action can or will be taken, the form must be signed by the analyst, supervisor, and QA.

3.4.8 Originals of all OOCE forms must be turned into QA. Copies must be included in each SDG or workorder in validatable packages and in the first workorder in the "samples affected" column for non-validatable data packages.

3.4.9 Any corrective actions taken which could either impact data directly, help to explain analytical decisions that were made in order to resolve analytical discrepancies, or which would help in the interpretation of the final data package must also be narrated in the final report. OOCE forms must be turned in with the data and the supervisor creating the narrative comment for that area will comment on any decisions resulting from failed QC which could impact data validity or interpretation.

Appendix I

Corrective Action Form

Laucks Testing Laboratories
Corrective Action Report

1) Problem Description:

Response tasked to: _____ on _____

By: _____ Response Requested By _____

2) Cause:

3) Action Taken:

Completed by _____ on _____

☐ Corrective actions will be reviewed 30 days after completion to verify problem has been corrected.

☐ No further action necessarv

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 CONTR 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)

Cold Storage T[illegible]rature Log

Laucks Testing L[illegible]ratories, Inc.

Cold Storage ID #:

Location: Year: 1996 Correction Factor (*add this number when* ***recording*** *the thermometer reading*): _____°C

Day	Month: Time	Temp.	Initials	Actions	Month: Time	Temp.	Initials	Actions	Month: Time	Temp.	Initials	Actions
1												
2												
3												
4												
5												
6												
7												
8												
9												
10												
11												
12												
13												
14												
15												
16												
17												
18												
19												
20												
21												
22												
23												
24												
25												
26												
27												
28												
29												
30												
31												

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

REFERFRM.DOC 5/29/96

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.

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1009

Title: **Blind Spike Program**

Revision history:

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

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

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

UNCONTROLLED

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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) Inorganic Statement of Work (SOW) and SW846.

1.1.3 Criteria for Precision and Accuracy (P&A) Studies are generally defined in the specific published method, particularly those in SW 846. Where criteria are not so defined, Laucks has chosen to either use the criteria from similar methods or to set in-house criteria based on the judgment of senior management and QA. Where two methods are the same in technical detail and one does not provide P&A criteria, performance under the guidance of the method with specifications may be used to satisfy the performance criteria of both.

1.1.4 Control limits are determined initially for an analysis, generally using limits supplied in the method or defined by the program (such as CLP). After sufficient points have been accumulated the laboratory performs a statistical analysis of the data and computes the control limits which are based on 3x the standard deviation of recoveries (for accuracy limits) or relative percent differences (for precision limits). In some instances, warning limits may also be established using 2x the appropriate standard deviation.

1.1.5 This SOP is designed for applicability to a wide variety of sample types ranging from reagent water to solids containing the analyte. The MDL may vary as a function of sample type. Laucks rarely determines MDLs on any matrix other than soil or water. Other MDLs may be estimated based on these studies.

1.1.6 This SOP requires that a specific, detailed analytical method exist. When determining MDLs and P&As following this SOP, it is imperative that all sample processing steps included in the analytical method be included.

1.1.7 Where a specific method has requirements exceeding the requirements of this SOP, that method will take precedence. Where a reference method has stated detection limits, these are generally taken to be MDLs. This SOP is to be followed to validate a new method or to validate a change in a current method.

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

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

1.1.10 This method is restricted to use by, or under the supervision of analysts experienced in the technique described. Each analyst performing this method must have demonstrated the ability to perform the described analysis except in the case of P&A studies which are used to demonstrate the competency of the analyst.

1.2 Method Description

1.2.1 Detection Limits

1.2.1.1 For any metals method, the Instrument Detection Limit (IDL) must first be determined. The IDL may also be determined strictly for informational purposes for other methods but is not required. The IDL allows the analyst to assess the precision of the measurement system and to estimate the target concentration for the MDL study. IDLs are generally determined by analyzing 7 low-level standard replicates on 3 non-consecutive days and averaging the sample standard deviations from each of the three days.

1.2.1.2 In order to determine MDLs, a minimum of seven replicate measurements are made of a prepared sample matrix which contains approximately 1 to 5 times the estimated detection limit. A Student's t determination is made for the number of data points available, usually 7 (6 degrees of freedom), and the resulting standard deviation multiplied by that value to determine the MDL. All MDL data are entered into the laboratory MDL database.

Note: The CFR states that the recommended concentration levels used to determine the MDL be one to five times the MDL. It later implies that a level of up to 10 times the MDL is acceptable. Laucks considers up to 10 times the MDL to be an appropriate concentration although limited exceptions to this rule may be granted as long as the deviations are not great and they are approved by QA.

1.2.1.3 Reporting Limits (RLs) are set by the laboratory as limits that can be reliably reported on a consistent basis with a reasonable degree of confidence that the reported level is accurate. These limits may be set at the Practical Quantitation Limit (PQL) initially by using a multiplier times the MDL. The multiplier is often but not always defined in the method. After initial setting of the RL, it is rarely changed unless significant changes in the MDL occur which make it necessary to raise or lower the RL.

1.2.2 Precision and Accuracy (P&A) Studies are studies performed in order to demonstrate the laboratory's ability to perform a method and are also used to demonstrate analyst competency to perform the method. They generally involve the analysis of 4 replicates spiked at concentrations defined in the method. Where no method guidance is provided, the replicates should be prepared at concentrations of 10 to 50 times the MDL for each analyte. Adequate performance is most often defined in the reference method, although if the method performance has been demonstrated, analyst competency may be demonstrated in comparison to laboratory limits.

1.2.3 Control limits may be specified in a reference method or may be statistically determined by the laboratory from existing data. In general, laboratory determined limits for control samples must not exceed method specified limits. If laboratory determined limits do exceed method-specified limits, the entire system must be evaluated to improve method performance. In most instances, it is unacceptable for routine performance to exceed method-specified performance even if the laboratory is using method-specified control limits. This is because the laboratory cannot demonstrate adequate performance for all samples on a routine basis.

1.2.4 It is not uncommon for clients to specify reporting or control limits in their project quality assurance plans. As long as they are achievable (i.e. the requested RL is not lower than the laboratory MDL), Laucks will generally comply with the client's request for that particular project.

1.3 Definition of Terms

1.3.1 **Accuracy** - The degree of agreement of a measurement (of an average of measurements of the same thing), X, with an accepted reference or "true" value, T, usually expressed as

the difference between the two values, X-T, or the difference as a percentage of the reference or true value, 100*(X-T)/T, and sometimes expressed as a ratio, X/T. Accuracy is a measure of the bias in the system. Accuracy shall be calculated as follows:

$$\%R = \frac{C_s - C_u}{S} * 100$$

Where:

Cs = Concentration of spiked sample
Cu = Concentration of unspiked sample
S = Expected concentration of spike in sample
%R = Percent recovery

1.3.2 **Control Limits** - Control limits may be specified in a reference Method (either as mandatory or guidance limits), or may be developed by the laboratory using internal performance data. Control limits represent acceptance criteria for determining whether an analytical system is in control (functioning within acceptable guidelines).

1.3.3 **Control Sample** - A QC sample introduced into the analytical process to allow evaluation of the measurement system. In general, it is best to use samples of a matrix similar to the samples being analyzed, where such are available. The control sample, however, will generally be free from interferences other than those inherent to the matrix itself.

1.3.4 **Degrees of Freedom** - The number of independent estimates that could be obtained from a specific set of data. In general, for a simple set of *n* independent values,

$$df = n\text{-}1$$

1.3.5 **IDL** - Instrument detection limit - The lowest concentration of a target analyte that can be measured and known to be greater than the instrumental background with a known degree of confidence. It may be used as a starting point for selecting MDL study spiking levels.

1.3.6 **MDL** - Method detection limit - The minimum concentration of a substance that can be measured and reported with a known degree of confidence (99% for our purposes) that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.

1.3.7 **Mean** - The arithmetic sum of a set of observations divided by the number of observations.

$$\frac{\sum X_i}{n}$$

Where:

X_i = sample value for replicate *i*
n is the number of replicates

1.3.8 **P & A** - Precision and Accuracy - This often refers to a study conducted to validate a method or an analyst conducting a particular method.

1.3.9 **PQL** - Practical Quantitation Limit - The limit at which it is determined that the constituent can not only be detected but be accurately quantified. This limit is usually 2 to 10 times the MDL but may be even larger depending upon the constituent and the matrix. Factors are often taken from the published method but may be set by the laboratory if published factors do not exist. These limits may also be used as the routine reporting limit (RL), unless otherwise contractually defined.

1.3.10 **Precision** - A measure of mutual agreement between individual measurements of the same property, usually under prescribed similar conditions. Precision is best expressed in terms of the standard deviation. Various measures of precision exist depending upon the "prescribed similar conditions".

1.3.11 **Reporting Limit (RL)** - A value greater than or equal to the MDL or the IDL which may be based on QA decision, the published method specifications, or project-specific requirements.

1.3.12 **Standard deviation** - A statistical measure of the variability of a set of sample observations. For the purposes of this SOP, the sample standard deviation is used. This is calculated using the formula:

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

Where:

s = the standard deviation estimated with n-1 degrees of freedom.
X_i = sample value for replicate *i*
$\overline{X}$ = mean of all of the replicates
n = the number of replicates

2. Equipment List and Standards

2.1 Equipment, Reagents and Standards

2.1.1 As appropriate for the given analysis.

2.1.2 Personal Computer with access to a spreadsheet program such as *Microsoft Excel* and the laboratory MDL database.

3. Safety precautions and Waste Disposal

3.1 Safety Precautions

3.1.1 Refer to the specific analytical SOP for appropriate safety precautions.

3.1.2 Waste Disposal

Refer to the specific analytical SOP for appropriate waste disposal practices. Waste segregation and disposal from the point of collection is further covered in the Laucks SOP on Waste Segregation and Disposal.

4. Calibration and Quality Control

4.1.1 Calibration is as appropriate to the specific method. No matrix spiking or other routine QA is required.

5. Responsibilities

5.1 Analyst

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

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

5.1.3 A metals analyst is responsible for assuring that a quarterly IDL study is produced on each instrument.

5.1.4 Each analyst is responsible for labeling MDL and P&A studies appropriately.

5.1.5 Each analyst is responsible for turning in a legible MDL, IDL, and P&A study to their supervisor for review and approval prior to final submittal to QA.

5.1.6 All of the analyst activities should be coordinated through the area supervisor.

5.2 Supervisor or Senior Analyst

5.2.1 Each area supervisor or senior analyst is responsible for coordinating the effective completion of the required studies. This may include but not necessarily be limited to helping determine appropriate concentration levels, coordinating the completion of the study within the timeline required by the method and/or the QA department, and scheduling the study around the analytical workload.

5.2.2 It is the responsibility of the area supervisor or senior analyst to insure that the analyst is performing the study within the guidelines of the method and to perform a review of the final data prior to submission to QA. This review should include determination that appropriate spiking levels were used, that the data was properly computed and transcribed, and that any problems or concerns encountered during the study are documented. Part of this review must include the comparison of the data to method specific criteria. In other words, P&A data must be compared to established method criteria and MDLs must be compared to Reporting Limits to ensure they are no greater than the RLs.

5.2.3 It is the responsibility of the area supervisor to obtain the necessary information to update the control limits at a minimum of annually. This may be done in conjunction with QA and the LIMS/MIS department.

5.3 QA Department

5.3.1 It is the responsibility of the QA department to issue a Corrective Action notice to any department who fails to turn in acceptable MDL, IDL, or P&A studies.

5.3.2 It is the responsibility of the QA department to work with supervisors to schedule studies and to maintain files of all current and historical studies.

5.3.3 QA will review and provide the final sign-off that the study meets requirements.

5.3.4 QA will review and provide the final sign-off of reporting limits.

5.3.5 QA will bear the responsibility to maintain the statistically determined control limits and to ensure that they are within those specified in the reference method.

6. Operation procedures

6.1 General

6.1.1 All studies must be given laboratory LIMS ID numbers. Although they may be initially stored in QA, they will eventually be moved into the laboratory filing system and must have identification numbers in order to be able to retrieve the raw data. Identification numbers will almost always be assigned by QA but in the absence of the QA Officer may be assigned by authorization of QA or the Laboratory Director. All studies will use the SAM client code QC_Officer in order to better track them at a later date.

6.2 Instrumental Detection Limits (IDLs)

6.2.1 It is rarely necessary to perform actual IDL studies except for metals analyses. For metals analyses, they are performed quarterly on each instrument. Studies may be useful, however, to demonstrate instrument capabilities and as a tool for estimating the Method Detection Limit (MDL). Although IDLs may be used as estimates to determine appropriate MDL spiking levels, it is strictly prohibited to compute the actual MDLs based on IDL determinations. The following guidelines are provided for several general class of analyses, regardless of whether an IDL is required for that analysis type.

6.2.2 As with all studies, a laboratory ID number should be assigned by QA for tracking purposes. In the case of metals IDLs, the same ID number may be assigned to all of the quarterly IDLs, rather than just one per instrument.

6.2.3 Actual IDLs studies are performed according to the CLP SOW by analyzing 7 replicates of low-level standards made up in the same matrix as all standards and not including any processing steps that would not ordinarily be performed on standards. The levels of those standards should be estimated from manufacturers detection limit specifications.

6.2.4 IDLs should be performed under the same instrumental conditions as will be used to perform actual analyses.

6.2.5 IDL studies must contain the following information (not necessarily in this order) for submittal to QA.

- Laboratory ID number
- Analyst who performed the IDL study
- Instrument name and ID which will distinctly identify that instrument
- Spike level
- Measured concentration of the 7 replicates (per day)
- Standard Deviation

- Mean
- Determined IDL
- Concentration Units
- Date(s) the study was analyzed
- Analysis (i.e. ICP, GFAA, etc.)
- Analysts signature & date signed
- Supervisor or senior analyst review signature & date signed

6.2.6 Spectrophotometry

6.2.6.1 The EPA/CLP SOW for metals requires that the IDL study be run on 3 non-consecutive days at least 7 times each day. It is prepared from an acidified aqueous standard solution made up at 3 to 5 times the manufacturers suggested IDL. The sample standard deviation (n-1) for each individual set of determinations is calculated and the final IDL is calculated as 3 times the average of the standard deviations for the three days. This may be performed using any commercial spreadsheet but care must be taken to insure that it is done using the sample standard deviation (n-1) calculation. For *Microsoft Excel*, this is the =STDEV() calculation. Ten percent of the calculations must be manually verified in order to demonstrate that the spreadsheet calculations are accurate.

6.2.6.2 If other spectrophotometric method IDLs are established by analyzing standards 7 times on 3 non-consecutive days, the calculation of the IDL is performed as described above. In addition, the EPA/CLP method does not prescribe the determination of MDLs. It is standard laboratory procedure to perform an MDL study (see section 6.3) approximately annually for almost all routine methods of analysis, regardless of IDL frequency or other determinations.

6.2.7 Chromatography

6.2.7.1 The analyst should use the signal:noise method for determining concentrations to use for an IDL study. A preliminary estimate of 5x signal:noise is to be used; if necessary this will be adjusted and the study repeated.

6.2.8 Gas Chromatography/Mass Spectrophotometry

6.2.8.1 Mass spectral identification criteria are key in selecting target concentrations for the IDL study. The mass spectroscopist's experience in determining the minimum identifiable concentration must weigh heavily in selecting concentrations. All compounds must meet the spectral matching characteristics as called out in the analytical method for the IDL study to be valid.

6.3 Method Detection Limits (MDLs)

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

6.3.2 MDL studies must also be performed when any major changes have been made in an instrument, such as a detector change.

6.3.3 Prior to beginning an MDL study, a laboratory workorder ID must be obtained from QA. The data generated from the study is then referenced to that workorder in the same manner as routine sample data.

6.3.4 MDL studies must contain the following information (not necessarily in this order). This will be accomplished by using the MDL database report plus an MDL Information Sheet (See Appendix 2).

- Laboratory ID number
- Analyst who performed the preparation
- Method number of the preparation (where applicable)
- Date(s) the study was prepared
- Method number of the clean-up (where applicable)
- Analyst who performed the MDL study
- Method number of the analysis
- Date(s) the study was analyzed
- Instrument name and ID which will distinctly identify that instrument; this cannot be a data "channel" from the computer system but must distinctly and uniquely identify that instrument.
- Spike level
- Measured concentration of the 7 replicates
- Standard Deviation
- Mean
- Determined MDL
- Concentration Units
- Reporting Limits (RLs)
- Analysts signature & date signed
- Supervisor or senior analyst review signature & date signed

6.3.5 The analyst must compare the MDLs with their current Reporting Limits (RLs) to ensure that they are no higher than the RLs. In fact, in most cases the MDLs should be demonstrably lower than the RLs unless there is a specific request to report down to the MDL.

6.3.6 If it is determined from the study that the reporting limits must be changed (i.e. the MDL is near to or exceeds the RL and cannot be re-determined with more appropriate results), the QA Officer and the supervisor, often in concert with the Laboratory and/or Technical Director(s), must meet to determine the appropriate course of action. Reporting limits are intended to be at a level for which method precision and accuracy can be obtained. This generally cannot be done when the RL is close to the MDL

6.3.7 In order to determine the Method Detection Limit (MDL), it is first necessary to estimate what the MDL will be in order that the appropriate spiking levels may be used. How this estimate is made is immaterial to the actual MDL determination. Methods for making this determination may include any one or a combination of the following:

- estimating based on the instrument detection limit (IDL) as determined above or by any other means
- estimating based on the previous MDL
- estimating based on 3 times the instrument signal to noise ratio
- estimating based on analyst judgment

6.3.8 A solution is then prepared and spiked into a sample matrix, which is as free as possible of interference and target analytes, at a level that will result in a sample concentration equivalent to 1 to 5 times the estimated MDL.

Note: The CFR states that the recommended concentration levels used to determine the MDL be one to five times the MDL. It later implies that a level of up to 10 times the MDL is acceptable. Although the analyst should make his/her best effort to spike at a level from 1 to 5 times the MDL, Laucks considers up to 10 times the MDL to be a sufficient concentration. Limited exceptions to this rule may be granted as long as the deviations are not great and they are approved by QA.

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

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

6.3.9 Preparation of Spiked Samples

6.3.9.1 The MDL is almost always determined in reagent water or clean sand. Prepare a laboratory standard containing all analytes of interest at a concentration which is at least equal to or in the same concentration range as the estimated MDL. The analyte concentration should not exceed 5x the estimated MDL but allowances may be made up to 10x the determined MDL.

6.3.9.2 It is extremely rare that Laucks will perform studies for other than reagent water or soil. Soil matrix will almost always be represented by clean blank sand except for metals analyses where even clean sand contains levels of some metals which exceed the 10x acceptance criteria. For such analyses, reagent spikes are used containing only the digestion/preparation reagents. MDLs on other matrices will generally only be performed upon specific client request.

6.3.10 Calculation of recovery statistics

Note: All values are used without correcting for native concentration. As previously mentioned, if blank correction is a part of the method, the average blank value is used for correcting analyte concentration measurements. In almost all methods, however, blank correction is forbidden.

6.3.10.1 The sample standard deviation is calculated as follows:

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

where:
s is the standard deviation estimated with n-1 degrees of freedom.
X_i = sample value for replicate *i*
$\overline{X}$ = mean of all of the replicates
n is the number of replicates

6.3.10.2 The Student's *t* statistic is determined for (*n* -1) degrees of freedom at the 99% confidence interval (CI). A Student's *t* table for the 99% CI is provided in Appendix 1. For most data sets, using n=7 sample readings, the *t* value is 3.143.

Note: In some cases, it may be determined that it is useful to prepare an additional sample so that, in case of laboratory accident, at least 7 are available for statistical analysis. Whether or not this is done, all samples analyzed **must** be used in the statistical evaluation unless there is a strong reason to reject one or more of the data sets, such as obvious contamination, abnormally poor surrogate recovery, set of values that are in obvious and significant disagreement with all of the others, or spilled sample. It is inappropriate to reject data which do not have an overriding reason to do so. The reason for rejection must be clearly documented in the data file. If more than 7 points are used in the MDL determination, the current MDL database will not accommodate the calculation. In this case, the determinations will necessarily be done using a spreadsheet program.

6.3.10.3 The MDL determination then becomes:

$$MDL = t_{99\%CI} * s$$

where:
$t_{99\%CI}$ = the Student's t value at the 99% confidence interval
s = the sample standard deviation as calculated above

6.3.10.4 The MDL, standard deviation and Student's t statistic for the appropriate number of replicates at the 99% CI are automatically calculated when using the Laucks MDL database.

6.3.11 Methodology Exceptions/Specifics

6.3.11.1 Wet Chemistry

6.3.11.2 The MDL for all titrimetric determinations is set as the value determined by 0.2 ml of titrant at the method specified titrant strength and sample aliquot size. This would include all tests such as versenate hardness, alkalinity, argentometric or mercurimetric chloride, titrimetric COD, etc. Karl-Fisher moistures would be an exception to this; the MDL is taken to the value determined by 0.05 ml of titrant, the method specified titrant strength, and sample size.

6.3.11.2.1 The MDL for all gravimetric residue determinations (total solids, total suspended solids, etc.) is set as the value determined by a weighing of 0.2 mg at the method specified sample size.

6.3.11.3 GC and Gas Chromatography/Mass Spectophotometry

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

6.3.11.3.2 Likewise, for PCB and other multi-peak GC analyses, pattern recognition may also dictate what can actually be determined. For either situation, analyst interpretation may be in order to confirm actual compound identification. Such interpretation must be noted in the data.

6.4 Reporting Limits

6.4.1 Reporting Limits are generally determined in one of four ways:

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

6.4.2 The administrative decision method is generally based on what the laboratory considers to be a limit which can be obtained on a consistent and reliable basis. Values obtained from statistical determinations of MDLs, for instance, cannot always be confirmed by spectral identification, pattern matching, standard response, or analytical spike recovery. In this instance, the laboratory may choose an RL which is more readily identifiable as a level for which a compound can be so identified and reliably quantified. Administrative decision may also be considered to be a part of the PQL option.

6.4.3 The PQL option is set as a factor times the MDL. This factor may either be set forth in the published method or it may be set by the laboratory. In order to be able to provide consistent and routine reporting limits, the laboratory will generally not reset PQLs when MDLs are re-determined unless the MDL changes by a factor of more than twofold.

6.4.3.1 If it is determined from the study that the reporting limits must be changed (i.e. the MDL is near to or exceeds the RL), the QA Officer and the supervisor, often in concert with the Laboratory Director and/or Technical Director, must meet to determine the appropriate course of action. Reporting limits are intended to be at a level for which reliable identification and reasonably accurate quantitation can be obtained. This generally cannot be done when the RL is close to the MDL.

6.4.4 Project Specific RLs are derived from project requirements and are contractually agreed upon between the laboratory and the client. In any event, the agreed upon limits **cannot** be less than the MDL or IDL.

6.4.5 On occasion, the low standard defines the RL. The decision to use this technique may be any combination of method specific requirements, laboratory decision, or project-specific requirements. In no case will the RL determined from the low standard be lower than the statistically determined MDL.

6.4.6 Reporting Limits are generally detailed in the Detection Limits Database and the LIMS system, unless set by project-specific agreement, in which case they are detailed in documents pertaining to that project and in the ProjQC database. The only persons given the capability to edit the approved limits are QA, LIMS system administrators, and the Technical or Laboratory Director. In most cases, only QA will actually perform any such editing. Note here that the EPA Contract Laboratory Program (CLP) requirements use specific contract required detection limits (CRDLs) or quantitation limits (CRQLs) and any project using the CLP methods will almost always also be reported using the CLP CRDLs or CRQLs. Any exception to the use of the CLP limits in these instances must also be noted in the ProjQC database and on any paperwork defining the details of the project.

6.5 Precision and Accuracy Studies

6.5.1 At a minimum, a one-time demonstration of precision and accuracy (P&A) must be performed for each method.

6.5.2 In some cases, it may also be required that an analyst will be required to perform a P&A study to be considered proficient and capable of independently performing a preparation or analysis.

6.5.3 P&A studies will be performed in accordance with the specific method. Where method-specific performance criteria are not specified, Laucks may choose to set criteria independently. Laucks' criteria, at a minimum, will meet those specified in a given method. Any determination to the contrary must be well documented and in direct consultation with QA and laboratory management.

6.5.4 All P&A studies must be turned in to QA after having undergone supervisory or senior analyst review.

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

- Laboratory ID number
- Analyst who performed the preparation
- Method number of the preparation
- Date(s) the study was prepared
- Analyst who performed the analysis portion of the P&A study
- Method number of the analysis
- Date(s) the study was analyzed
- Instrument name and ID which will distinctly identify that instrument; this cannot be a data "channel" from the computer system but must distinctly and uniquely identify that instrument.
- Spike level
- Measured concentration of the 4 replicates
- Standard Deviation of the recovery tabulated against the published QA Acceptance Criteria Table, where available
- Average recovery tabulated against the published QA Acceptance Criteria Table
- Concentration Units
- Analysts signature & date signed
- Supervisor or senior analyst review signature & date signed
- Raw Data

6.5.6 The mean recovery and acceptance limits must meet the criteria given in the QC Acceptance Criteria Table at the end of each of the determinative methods, when available. Where criteria are not available Laucks may use internal acceptance criteria or defer to a similar technical method with P&A criteria and use this P&A criteria as guidance in establishing performance criteria. In the case of organic SW846 methods, if the criteria are not published in the individual method, the criteria in method 8000 (70%-130%) are followed as a guidance. In many instances, 70-130% is not achievable on a routine basis even by skilled staff. In this case, the laboratory (senior staff in conjunction with QA) may determine its own acceptance limits.

6.5.7 Blank spike analyses are the commonly accepted P&A evaluation. In most methods where criteria are defined, 4 replicates must meet method-specified criteria for the **laboratory** to be considered capable of adequate performance.

6.5.8 The individual **analyst** must be able to analyze four replicates and meet laboratory blank spike control limits to be considered competent to perform the applicable analysis. For purposes of the P&A study, the analyst may be considered qualified if 90% of the analytes in a multi-analyte analysis meet laboratory criteria as long as all analytes meet the default method-specific criteria.

6.5.9 For the laboratory to be able to claim routine performance within specified limits, all analysts performing an analysis must be capable of that level of performance. All analysts must be routinely capable of performance within method-specified criteria and will be evaluated against laboratory criteria, with further action and training in order if they are unable to routinely meet laboratory criteria.

6.6 Control Limits

6.6.1 Initially, when a new method is being implemented or there are insufficient data, the laboratory will use method-specified control limits for evaluation of data. If no such limits exist, the laboratory may elect to use specified limits from a similar method or may set default limits at the laboratory's discretion. These limits may be from the precision and accuracy study for that method. The determination for the suitability of setting any default limits not otherwise specified in a reference method is at the discretion of QA.

6.6.2 During the routine course of analysis, blank spike or laboratory control samples (LCS) and in many cases matrix spikes and matrix spike duplicates (or sample duplicates) will be analyzed. Spiking will occur at the levels specified in the respective methods where available, but will generally be somewhere in the middle of the calibration range.

6.6.3 When sufficient data have been gathered, generally at least 20 data points, the laboratory will undertake the determination of statistically-based control limits. These control limits are based on 3x the standard deviation of recoveries (for accuracy limits) or relative percent differences (for precision limits). In some instances, warning limits may also be established using 2x the appropriate standard deviation.

6.6.4 At a minimum, the control limits will be updated annually on a preparation/analysis/matrix specific basis. The number of data points and spiking levels used to obtain the new limits must be documented when forwarded to QA for approval.

6.6.5 If purchased from a commercial vendor, vendor-supplied control limits for a control sample will be considered adequate for default control limits if they are within the limits specified in the reference method. In addition, if the material is readily available and its composition does not change with every purchase, the laboratory will develop internal limits for that material. These limits may or may not be within the vendor-supplied limits but they **must** be within the method-specified limits.

6.6.6 In general, laboratory determined limits for **control samples** must not exceed method specified limits. If laboratory determined limits do exceed method-specified limits, the entire system must be evaluated to improve method performance. In most instances, it is unacceptable for routine performance to exceed method-specified performance even if the laboratory is using method-specified control limits. This is because even though the

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

6.6.7 The laboratory may also calculate limits for matrix spike and matrix spike duplicate or replicate samples. However, these limits are primarily used to demonstrate method performance on a particular sample or sample-type relative to the routine laboratory sample and exceptions to these limits will generally be allowed as long as control sample limits are met.

6.6.8 The laboratory may be called upon to utilize control limits specified in a method or in a specific contract as designated in the LIMS ProjQC database or supplementary paperwork. The laboratory's overall performance will be considered adequate if internal control limits are within those specified in the reference method. Contractually defined limits will be used for the control samples analyzed under the contract and appropriate corrective actions taken but will not be used as a guide for routine laboratory performance.

6.6.9 For any particular project, if the laboratory exhibits exceptions to the method or contract-specified criteria, appropriate corrective action must be taken. Should routine laboratory control limits be within method or contract-specified criteria, and laboratory limits are exceeded but method or contract limits are met, the data may be reported but should be flagged. Where appropriate, corrective action may still be taken at the discretion of QA.

7. Reports

7.1 Data Package Organization

7.1.1 All work, with the exception of control limit computations, is performed under laboratory workorder ID numbers.

7.1.2 All data supporting the study are provided in a standard format specific to that method. In order to save paper, some items, such as the initial calibration, etc., may be referenced to other workorders. However, it must all be easily recoverable if full documentation is required, up until the standard laboratory data disposal date. Rationalizations for interpreting the results of any study and specific detail which might impact the study should be documented in the file as well.

7.1.2.1 Data files are prefaced with a copy of the summary report containing all of the elements previously noted in this SOP. Where laboratory database reports are available, a copy of the database report must also kept on file by QA. All sign-offs will be handwritten.

8. References

40 CFR Part 136, Appendix B, *Definition and Procedure for the Determination of the Method Detection Limit, Revision 1.11*

EPA Contract Laboratory Program (CLP) Inorganics Statement of Work (SOW), ILM04.0.

EPA "600" Series Methods, section 8.1.1, 40 CFR, Part 136, App. A.

EPA SW846 "8000" Series Methods, both the general method 8000B and the specific methods

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

Appendix I

Student's *t* Values

n	degrees of freedom	*t* value at 99% CI
2	1	31.821
3	2	6.965
4	3	4.541
5	4	3.747
6	5	3.365
7	**6**	**3.143**
8	7	2.998
9	8	2.896
10	9	2.821
11	10	2.764
12	11	2.718
13	12	2.681
14	13	2.650
15	14	2.624
16	15	2.602
17	16	2.583
18	17	2.567
19	18	2.552
20	19	2.539
21	20	2.528
22	21	2.518
23	22	2.508
24	23	2.500
25	24	2.492

Appendix II

MDL Information Sheet

Laucks Testing Laboratories, Inc.
MDL Information Sheet

This form must be submitted in addition to the information supplied on the SAM MDL summary form along with the supporting raw data.

SAM MDL Name: _____

SAM Workorder Number: _____

Analysis:

Analyst: _____

Analysis Method: _____

Analysis Date: _____

Instrument ID: _____

Preparation:

Prepared by: _____

Preparation Method: _____

Preparation Date: _____

Cleanup Method(s): _____

Review:

Analyst's Signature: _____

Supervisor's Signature: _____

QA Approval: _____

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #: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

UNCONTROLLED

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1. Introduction and Scope

1.1 Method Description

1.1.1 This SOP is intended to describe the way in which standards and reference materials are tracked, prepared, stored and maintained at Laucks, from the time of receipt of the neat or stock materials, solutions or their preparation to the point of use of the working standard. General descriptions of documentation of standard preparation may be present, it is not intended to define the actual method of preparation for each specific method. This is contained in the applicable analytical method SOP. The way in which these standards are tracked, however, is detailed along with the description of storage and shelf life guidance.

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

1.2 Definition of Terms

1.2.1 Standard or Reference Material: these items are defined as any solution of an analyte at a known concentration prepared from purchased neat materials or stock solutions, or from intermediate solutions traceable to purchased materials. This includes calibration standards, independent laboratory control standards (LCS or SRM), spiking solutions, surrogate solutions, independent calibration verification standards.

2. Equipment Lists and Standards

2.1 Equipment

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

2.2 Standards

2.2.1 Standards as specified in each analytical SOP.

2.2.2 All standards must also be verified both qualitatively and quantitatively in order to satisfy EPA requirements for traceability. This may be accomplished by either (1) purchasing solutions which have been fully documented by a commercial vendor, or (2) following the recommended steps for traceability as outlined in the 3/90 CLP Organic statement of work.

2.3 Standards Logbooks

3. Safety precautions and Waste Disposal

3.1 Safety Precautions

3.1.1 All standards and reference materials including neats or solutions should be handled as if they are hazardous substances.

3.2 Waste Disposal

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

4. Operation Procedures

4.1 Preparation of Organics and Inorganics Materials

4.1.1 General consideration in standard preparations include:

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

4.1.1.2 Choose volumes and aliquots which minimize the number of intermediate dilutions required to obtain final working concentration considering:

- The inherent measurement error, i.e. no aliquots less than 20% of the volume of measurement device whenever possible.
- The ratio of solvent:analyte
- The amount of solution left over for disposal.

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

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

4.1.2 Proper Syringe/Pipette Technique

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

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

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

4.1.2.4 There should be no air bubbles. Either tap them away or discard the solution in the syringe/pipette and obtain another aliquot. Repeat this procedure as often as necessary to remove all bubbles. It may be helpful to use a GC septum with very small (<50 µl) syringes.

4.1.2.5 For organics, when delivering the measured volume to the dilution vessel, fill the vessel 1/2 - 2/3 with the solvent to be used, add the measured aliquot directly into the solvent without touching the sides of the container, and fill to volume with solvent. A sub-surface injection is preferable whenever possible.

4.1.3 When preparing stock solutions from neats, the following steps should be taken.

NOTE: 99.9% of the time, stock standards will be prepared WEIGHT per Volume. DO NOT use Volume measurements for liquids unless EXPRESSLY TOLD to do so by your SUPERVISOR.

4.1.3.1 The dilution vessel (volumetric flask) and stopper should be triple solvent rinsed (last time with the solvent to be used for standard preparation) and allowed to dry completely.

4.1.3.2 The neat is weighed, to 4 significant figures, directly into the volumetric flask and the weight is recorded (to 3 decimal places for volatiles, one less than actually weighed in order to account for possible small losses due to volatilization). Stopper before weighing to avoid compound volatilization if dealing with solvents or volatile materials.

4.1.3.3 For components other than volatiles, the volumetric flask is filled about 3/4 full with dilution solvent and shaken until analyte is completely in solution.

- If the analyte will not dissolve, the stoppered volumetric flask should be sonicated in the sonic bath until it does dissolve. (Because sonication heats the solution slightly, the solution should be allowed to cool before dilution to the

mark). Consult your supervisor if the compound is not in solution after sonication.

- The volumetric flask is diluted to the mark.
- If the analyte recrystallizes while stored in the refrigerator, the standard should be sonicated before use. Do not aliquot from a cloudy or opaque standard.
- In addition to the normal labeling of the standard, a separate label should be added indicating the need for sonication.

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

4.2 Traceability Documentation for Organics and Inorganics Materials

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

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

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

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

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

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

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

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

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

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

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

4.2.6 After each material is logged it is labeled with the LAUCKS ID, date received, date opened (if the material is to be used from the same container more than once) and expiration date (if not already on the label). The accompanying vendor Certificates of Analysis, Purity or Authenticity are labeled with the Laucks ID and filed in a controlled laboratory notebook in the laboratory area. These certificates are then archived through QA when the notebook is full.

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

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

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

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

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

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

4.2.10 The Laucks internal working material ID must be documented on the manual benchsheet, the analytical run-log or instrument printout to enable tracking back to the parent material. See Appendix 5 for examples of typical bench sheets with standards references.

4.3 Storage of Standards and Reference Materials

4.3.1 Always completely label solution with the following information:

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

4.3.2 Organic Standards and References Materials

4.3.2.1 Store in vial or bottle which minimizes head space.

4.3.2.2 Use amber or clear glass, screw tops with Teflon-liners when required, and store at, in order of preference, the temperature referenced in the analytical SOP or the temperature detailed below, in the assigned refrigerator.

4.3.2.3 Volatile Standards and Reference Materials

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

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

4.3.2.3.3 Standards are transferred to Mininert vials with Teflon lined septa for daily use and stored in the VOA freezer. When the standards are transferred, the information is recorded in the GC/MS Volatile Standards log book.

4.3.2.4 Other Volatile Standard Solutions

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

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

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

4.3.2.4.4 Cover ends of ampules with foil.

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

4.3.2.4.6 Mix gently.

4.3.2.4.7 Partially fill ampules with solution and recap with foil.

4.3.2.4.8 Use CO_2 to cool ampules until crystals form on sides.

4.3.2.4.9 Heat end of ampule with acetylene flame until glass begins to soften.

4.3.2.4.10 Gently pull end until seal is formed.

4.3.2.4.11 Label ampules and store in freezer.

4.3.2.4.12 Record the information in the Mass Spec VOA Standards Log Book (MV).

4.3.2.4.13 When standard solutions are used they should be transferred to Mininert cap vials with Teflon lined septa. The vials are stored in the VOA freezer until discarded.

4.3.2.5 Semivolatile Standards and Reference Materials

4.3.2.5.1 All standards solutions should be stored at a maximum temperature of 4 degrees C (± 2 degrees). Refer to the analytical SOPs for details as some analytes may drop out of solution if at cooler temperatures.

4.3.3 Inorganic Standards and Reference Materials

4.3.3.1 All metals standards are kept in a cabinet in the metals analysis lab. This is at room temperature. Expired standards that are kept for qualitative purposes are kept in the same room, in a different cabinet. These qualitative standards have a special label on the bottles denoting that they are not to be used for quantitative purposes. All other standards are kept at 4°C in a reach-in cooler in the inorganics lab. This cooler is dedicated to standards and SRMs only. No sample storage is allowed in this cooler.

4.4 Shelf Life

4.4.1 Expiration

4.4.1.1 If a parent material has an expiration date of month/year, then the material is considered usable through the end of that month. For example, 01/96, the material expires after 1/31/96. This guidance was obtained from various vendors.

4.4.1.2 All parent expiration dates MUST be entered into the standard log books and the expiration date for all resulting child materials must also be entered into the logbook and placed on the material label.

4.4.1.3 Note that no child solution may exceed the life of a parent solution or neat material. This stipulation may reduce the shelf life of a prepared solution from that listed in Appendix 2. For instance, if a stock solution is prepared from parent material that has an expiration date of 05/20/95 in 01/95, instead of having a six month shelf life (07/95) the solution will expire, 05/20/95, the same date as the parent.

4.4.1.4 See Appendix 2 for the Table of typical shelf life of standards and reference materials. This table is provided as guidance only. The vendor expiration date (if applicable) and the analytical SOP take precedence over any guidance set forth in the Table.

4.4.1.5 If a standard is past its expiration date it may be used for qualitative purposes only. The standards logbook must be edited to reflect this status and an additional label must be placed on the standard. This label must be bright in color and must clear indicate that it is to be "Used for Qualitative Purposes Only".

5. Standard Verification

5.1 Criteria

5.1.1 Standards are to have their concentrations verified before use whenever possible. The QC'ing of the standard is to be recorded in the applicable column in the standards logbook unless they are validated in the individual analytical run (such as confirmation by another standard from an independent source). Criteria for standards acceptability are in many cases defined in individual SOPs. In instances where they are not so defined, acceptability criteria are:

- 80% - 120% for organics
- 90% - 110% for inorganics

Appendix 1

Example Calculations

1. Concentration Calculations from Neat Materials

HELPFUL hint: To keep yourself straight ALWAYS, ALWAYS include the units (mg, ml, etc.) in your calculations.

Example Calculations of Standard Concentrations:

Weight of Neat Material:	0.2500 gm
Volume of Solvent:	10 ml

To Calculate Concentration in mg/L (ppm):

1) Calculation in Steps.

A) $$0.2500gm * \frac{1000mg}{1.0g} = 250mg$$

A.1) Adjust the 250 mg for purity,

i.e. if purity = 90%, 250 mg x 0.9 = 225 mg

B $$10mls * \frac{1L}{1000mls} = 0.01L$$

C) $$\frac{225mg}{0.01L} = 22500mg / L$$

2) Calculation as a Single Step.

$$\frac{0.2500gm}{10ml} * 0.90(purity) * \frac{1000mg}{1gm} * \frac{1000ml}{1L} = 22500mg / L$$

3)

$$FC = \frac{W}{FV} * P * \text{Conversion Factors}$$

where;

W = Weight of neat material (g)
FV = Final Volume (ml)
P = Purity (%/100)
FC = Final Concentration (mg/L = ppm)

2. Intermediate and Working Standards (Standard Dilution)

$$(FC)(FV) \times 1000 = (AV)(PC)$$

where;

FC: Final Concentration(s) in standard desired. Units=µg/mL.
FV: Final volume of the prepared standard. Units=mL.
1000: Conversion factor from mL to µL
PC: Parent Concentration (standard normally containing high concentrations and is diluted to desired final concentration). Units = µg/mL.
AV: Aliquot Volume of parent standard required to achieve final concentrations desired.
Units: µL (microliter).

a) Neats to Stocks

$$\frac{Purity * 1{,}000{,}000 * W}{FV} = FC$$

where;

1,000,000 = Conversion factor from gram to microgram
W Weight used in standard prep (g)
FV Final Volume (ml)
FC µg/ml = ppm = mg/L
Purity = % Purity/100

For Example: 100% = purity of 1.0
86% = purity of 0.86

If the % purity is ≥ 97%, it is considered 100% pure for standards calculation.

Appendix 2

Shelf Life Guidelines

NOTE: *IN NO CASE, will the Laucks' expirations date EXCEED the manufacturer's expiration date.
IN NO CASE, will a child solution have an expiration date that exceeds its parents.

TYPE OF STANDARD	INORGANICS	ORGANICS EXTRACTIONS	ORGANICS INSTRUMENTATION[A]
Purchased Neat	10 Years *[1]	5 Years *[1]	5 Years *[1]
Purchased Stock Solution	12 Months *[2]	12 Months *[2]	6 Months *[2]
Prepared Stock Solution	12 Months	12 Months	6 Months
Intermediate Solution	3 Months	N/A	6 Months
Working Solution	2 weeks *[3]	6 Months	3 Months
Purchased Working Solution		6 Months	3 Months

* 1. Unless the manufacturers expiration date is less than the following, purchased neat standards shelf life will not exceed 10 years materials from the date of receipt for inorganics and 5 years from the date of receipt or 3 years from the date opened for organic materials, whichever is shorter.

*2. Unless manufacturers expiration date is less than the following, purchased stock solutions or intermediates shelf life will not exceed 1 year from the date opened.

* 3. Listed time is maximum. Specific shelf-life criteria are detailed in the individual SOPs.

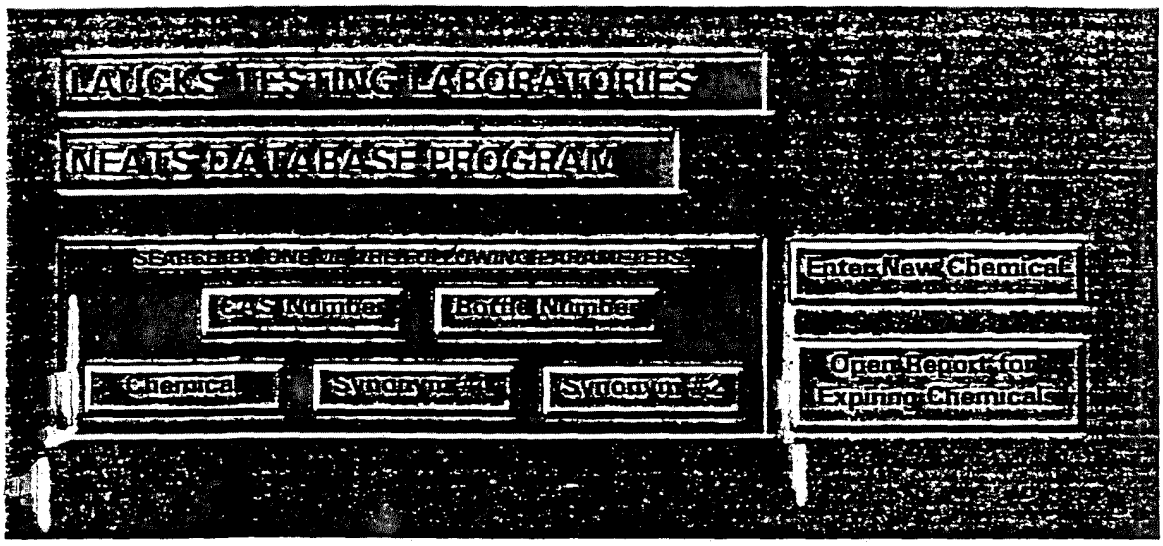
NOTE: THIS IS A GENERAL PROTOCOL. WHERE POSSIBLE, VERIFY THE INTEGRITY OF THE WORKING STANDARD SOLUTION BY MEETING THE ACCEPTANCE CRITERIA SPECIFIED IN THE ANALYTICAL SOP FROM THE KNOWN TRUE VALUE WHEN ANALYZED AGAINST AN INDEPENDENT LABORATORY CONTROL STANDARD OR A CALIBRATION CURVE.

A. Volatiles:

Method	Expiration Date
SW 846 8240 B	Stock Standards: 6 months; gases weekly if unstable, or 6 months if prepared in nitrogen. Calibration standards prepared daily
CLP OLM01.9	Stock Standards: 6 months or sooner. Stock gas standards: 2 months or sooner Secondary dilution standards: 6 months or sooner (gases & reactive compounds: monthly or sooner) Calibration standards: weekly or sooner. IS, surrogate & matrix spike: fresh spiking solution weekly or sooner. Aqueous standards: 24 hours at 4°C or 1 hour at room temperature; 12 hours if stored on autosampler.
CLP OLM0 2.0-03.1	Stock Standards: 6 months or sooner. Gases & reactive compounds: 2 months Secondary standards: 1 month or sooner for gases & reactive compounds, e.g. styrene Other purgeables: 6 months or sooner IS, surrogate & matrix spike: fresh spiking solution weekly or sooner. Calibration standards: weekly or sooner. Standard solutions stored in ampulated glass vials for 2 years from preparation date or shorter if recommended by manufacturer. Once opened, expiration dates above apply. Aqueous standards: 24 hours at 4°C or 1 hour at room temperature; 12 hours if stored on autosampler.
10/92 Low Conc. CLP	Opened stock standards: weekly Aqueous standards: 24 hours. Stock Standards: 6 months or sooner Gases stock standards: 2 months or sooner Secondary dilution standards: 6 months or sooner (gases 1 month or sooner) Working calibration standards: weekly. IS: prepare fresh spiking solution every 3 months or sooner Surrogates: prepare fresh surrogate solution every 6 months or sooner
SW 846 8260A	Stock Standards: 6 months or sooner Gases: weekly if unstable or 6 months if prepared in nitrogen Working solutions: check frequently for degradation or evaporation Calibration standards are prepared daily

Appendix 3

Neats Database Screens


LAUCKS TESTING LABORATORIES
NEATS DATABASE PROGRAM
CAS Number
Bottle Number
Chemical
Enter New Chemical
Open Report for Expiring Chemicals

NEATS DATA ENTRY

Chemical:	n-nitroso-diphenylamine	CAS #:	86-30-6
Synonym #1:		Purity (%):	98.3
Synonym #2:		Lot #:	153-150B
Bottle #:	N167[illegible]	Source:	ChemService
Nr Bottles:		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

Appendix 4

Logbook Examples

LAUCKS TESTING LABORATORIES, INC.
STANDARDS LOGBOOK

#	ANALYTE	ID or STOCK No.	STOCK CONC.	VOL/WT TAKEN	FINAL VOL.	CONC.	SOLVENT	PREP. DATE	INIT.	EXP. DATE	QC'D (Initials)
1	SW846 high std	ME3-96-01 10/19/96	LTL-CAL-1	10 mls	100 mls		HCL 5% HNO3 1% H2O	~~4/3/96~~ KP 4/2/96	KP	7/10/96	KP
		ME4-45-01 4/1/97	LTL-CAL-2	1 ml							
		ME4-3~~4~~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 EKP 5/96	1000ppm	100 µl	5-11-96 ~~[illegible]~~ 100mL	1ppm	5% HNO3	4-3-96	tm	5-96	
4	ICP/MS STD1	ME4-30-02	ICP/MS	500 µl	5mL		5% HCL H2O	~~4/3/96~~ 4 ABN	ABN		
5	STD2		Starch Std	250							
6	STD3			500							

ME4-48

LAUCKS TESTING LABORATORIES, INC.
STANDARDS LOGBOOK

#	ANALYTE	ID or STOCK No.	STOCK CONC.	VOL/WT TAKEN	FINAL VOL.	CONC.	SOLVENT	PREP. DATE	INIT.	EXP. DATE	QCD (Initials)
1114-03-04 con't 4	2,4,6-TNT	M-8330-11	100 ppm	10 ul	50 ml	200 ppb	DI H2O	12/20/95	MH	04/01/96	
↓	HMX	M-8330-4	↓	↓	↓	↓	↓	↓	↓	↓	
01	1,3,5-TNB	M-8330-12	1000 ppm	10 ul	50 ml	200 ppb	Fluid TCLP #1	12/20/95	MH	04/01/96	
	2,4-DNT	M-8330-2									
	RDX	M-8330-5									
	2,4,6-TNT	M-8330-11									
↓	HMX	M-8330-4					↓				
02	1,3,5-TNB	M-8330-12					TCLP #2 Fluid				
	2,4,-DNT	M-8330-2									
	RDX	M-8330-5									
	2,4,6-TNT	M-8330-11									
↓	HMX	M-8330-4	↓	↓	↓	↓	↓	↓	↓	↓	

LAUCKS TESTING LABORATORIES, INC.
STANDARDS LOGBOOK

#	ANALYTE	ID or STOCK No.	STOCK CONC.	VOL/WT TAKEN	FINAL VOL.	CONC.	SOLVENT	PREP. DATE	INIT.	EXP. DATE	QC'D (Initials)
1	IC ICV Set SK 3/11/96 Cl	ERA 3412	—	—	250ml	129 mg/L	—	logged in 3/18/1996	SK	Opened: 4/8/96 Exp: 4/19/97	
	F		—	—		6.47 mg/L	—				
	NO_3		—	—		8.77 mg/L	—				
	SO_4		—	—		11a mg/L	—				
2	Cal. Std. F	IC4.84.4	1000 ppb	500 µl	100ml	5 ppb	H_2O	3/20/96	SK	4/3/96	
	Cl	.5	1000 ppb	1000 µl		10 ppb					
	NO_2	.7	100 ppb	2000 µl		2 ppb					
	NO_3	.3	1000 ppb	500 µl		5 ppb ~~10 ppb~~ SK 4/20/96					
	SO_4	.6	1000 ppb	2000 µl		20 ppb					
3	NO_2 ICV	IC4.78.7	100 ppb	1000 µl	100ml	1 ppb	H_2O	3/20/96	SK	3/27/96	
4	NO_2 ICV	IC4.78.7	100 ppb	1000 µl	100ml	1 ppb	H_2O	3/25/96	SK	4/1/96	

Appendix 5

Bench Sheet Examples

As, Tl

utoSampler Report Table: SW846 Wed 04-10-96 09:39:45 AM page 1

Name: SW846 Autosampler Type: TYPE TJA
Positions: 257/300 QC Positions: 11/19 # Sets: 1
inse Station location is rack -1, pos. -1.

-- Racks ---

ack #	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 ---

et#	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

et#	Uptake	Uptake#2	Final	Dil.Factor

lo Samples Prepared.

#1

Pos	Row	Col	Sample Name	Set #	#Used	Type
1	1	1	ICV1 ME4-48-01	-NA-	1	QC Standard
2	1	2	STD4 ME4-48-01	-NA-	1	Standard
3	1	3	STD3 ME4-48-01 5mls/10mls	-NA-	1	Standard
4	1	4	STD2 ME4-48-01 1ml/10mls	-NA-	1	Standard
5	1	5	STD1 ME4-45-02	-NA-	1	Standard
6	1	6	STD0	-NA-	1	Standard
7	1	7	Blank	-NA-	7	Blank
8	1	8	CCV ME4-51-01	-NA-	6	QC Standard

(9...19 Not Used)

Rack #2

Pos	Row	Col	Sample Name	Set #	#Used	Type
1	1	1	CRII1 ME4-45-02	1	-NA-	Sample
2	1	2	ICSABI1 ME4-52-05	1	-NA-	Sample
3	1	3	PBS1	1	-NA-	Sample
4	1	4	LCSS1	1	-NA-	Sample
5	1	5	03040-01	1	-NA-	Sample
6	1	6	03040-01D	1	-NA-	Sample
7	1	7	03040-01S	1	-NA-	Sample
	1	8	03040-01L	1	-NA-	Sample
9	1	9	03040-02	1	-NA-	Sample
10	1	10	03040-03	1	-NA-	Sample
11	1	11	03040-01 5X	1	-NA-	Sample
12	1	12	03040-01D 5X	1	-NA-	Sample
13	1	13	03040-01S 5X	1	-NA-	Sample

LAUCKS TESTING LABORATORIES
ABN GC/MS OPERATIONS LOG

CASE # _____ PAGE 4883 -A

3/18/96 IS = MS334-1 DFTPP = MS334-4

RUN #	FILE NAME	CLIENT NAME	CLIENT SAMPLE#	LAB I.D. #	INJECTION INFO	IS A RT	IS A RESPONSE	IS B RT	IS B RESPONSE	IS C RT	IS C RESPONSE
1	MH7501										
2	HC18A	—	SSTD050	MS4-9-1	25.% ABN	11.24	55075	14.99	159300	20.44	104485
3	HC18B	—	SSTD200	MS3-89-1	100.% only phenol	11.22	59100	14.98	169403	20.42	100469
4	HC180	Kalama	B031396nsmwlk	9603368-BLK	1000→1	11.23	58139	14.98	166207	20.43	99304
5	HC181	Kalama	S031396nsmwlk	—SPK	1000→1	11.24	55723	14.99	155289	20.44	98643
6	HC182	Olympia	—	9603206-01DL	1:4 1000→1	11.24	52364	14.98	158592	20.44	102071
7	HC183	Kalama	—	9603368-01	1:2 100→1	11.24	58997	14.98	168568	20.46	103091
8	HC184	↓	—	-01DL	1:4 100→1	11.24	58386	3/3/96 14 15.00	173180	20.45	106213

3/19/96

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1017

Title: Internal Audit Procedures

Revision history:

Number	Date
000	5/13/93
001	3/3/96

Written by: Harry Romberg Date: 3/3/96
Harry Romberg

Approved by: Karen T. Kotz Date: 3/3/96
Karen T. Kotz, Laboratory Director

UNCONTROLLED

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1. Introduction and Scope

1.1. Method Description

1.1.1. The purpose of this procedure is to provide instructions for planning, performing and reporting QA/QC audits within the laboratory.

1.1.2. This method is restricted to use by, or under the supervision of personnel experienced in the technique described.

1.2. Discussion

An Audit of the facility is performed for the following reasons:

1.2.1 To determine that contractual and regulatory obligations are fulfilled.

1.2.2 To determine that procedures and standards are being followed, and to insure good laboratory practice. These audits will include, but are not limited to the refrigeration unit temperatures, logbooks, balance calibrations, data, and standards traceability.

1.2.3 To establish that quality assurance objectives are met, including holding times, use of approved analytical methods, and stated objectives for precision and accuracy.

1.2.4 To serve as a management tool to evaluate appropriateness of quality assurance policies.

1.2.5 To identify potential or actual deficiencies for the purposes of evaluating compliance with requirements and providing the means for correction.

1.2.6 To determine that records are prepared and maintained as required.

1.3 Documentation and Frequency

Documentation required is specified in the text and the frequency shall be as required by the QA Manager, but at least one technical audit shall be performed annually for each department. This audit may take place in parts, with additional and more extensive audits being scheduled as deemed necessary.

1.4. Definition of Terms

1.4.3 This section defines terms and acronyms as they are used in this SOP.

1.4.4 SOP: Standard Operating Procedure

1.4.5 QA: Quality Assurance

1.4.6 QC: Quality Control

1.4.7 Audit: A planned and documented activity performed to determine by investigation, examination, or evaluation of objective evidence the adequacy of and compliance with established procedure, instruction, and other applicable documents and the effectiveness of implementation. An audit should not be confused with surveillance or inspection activities performed for the sole purpose of process control or product acceptance.

1.4.8 Auditor: Any individual: who performs or assists in the performance of any part of an audit, including technical specialists.

1.4.9 Lead Auditor: An individual who is qualified to organize and direct an audit, report audit findings, and evaluate proposed corrective actions.

1.4.10 Finding: Departure from approved procedures, program requirements, or other applicable documents that have, or in the immediate future could reasonably be expected to have, an adverse effect on the adequacy or effective implementation of the Laucks QA program. This would be ranked as a **critical** discrepancy in the audit report.

1.4.11 Deficiency: Departure from approved procedures, program requirements, other applicable documents, or good management practices that, if not corrected in a timely manner, could reasonably be expected to have a future adverse effect on the adequacy or effective implementation of the Laucks QA program. This would be ranked as a **minor** discrepancy in the audit report.

1.4.12 Discrepancy: Departure from approved procedures, program requirement, or other applicable documents that have, or may have an adverse effect on the adequacy or effective implementation of the Laucks QA program. This includes findings and deficiencies found during the course of an audit.

1.4.13 Recommendation: An observation or advise given to enhance current practices by any individual or department of the Laucks QA program. This would be ranked as a **recommended** item in the audit report.

2. Responsibilities

2.1 It is the responsibility of QA personnel. the auditor and audit leader to perform an audit according to this SOP and complete all documentation required for review.

2.1.1 QA Manager is responsible for the following:

- Approving each detailed audit plan
- Concurring with the adequacy of each audit report
- Issuing the audit report
- Tracking audit status through final closeout

2.1.2 If an audit team is used. the following responsibilities fall upon the Audit Team Leader. If an audit team is not used. the following responsibilities fall to the QA Manager:

- Developing the detailed audit plan
- Conducting pre-audit and post-audit conferences
- Supervising the conduct of the audit
- Preparing and signing the audit report

2.1.3 Management of audited departments is responsible for the following:

- Providing reasonable and timely access to personnel, facilities, and records, as required to support the audit process
- Providing timely and adequate response to audit reports, including determination and implementation of corrective actions. as required.
- Verifying initial implementation of corrective action for deficiencies in their areas, if applicable.

2.2 Audits and reports are to be performed by personnel in the laboratory who have demonstrated the ability to evaluate processes in the laboratory with emphasis on Quality Control and Quality Assurance.

2.3 Final review and sign-off of each Audit Finding Report may be performed by either the QA Manager. Lab Director or department supervisor or designee.

3. Safety precautions

3.1. Safety Precautions

3.1.1. Auditors must adhere to the general laboratory health and safety policies during the course of the audit.

3.1.2 Protective eyewear must be worn in all applicable locations at all times during the course of the audit.

4. Calibration and Quality Control

Not applicable.

5. Operation procedures

5.1 General

5.1.1 Audit personnel may be selected and assigned audit responsibilities commensurate with their training and expertise and the special nature of the activities to be audited.

5.1.2 Audit personnel are independent of any direct responsibility for performance of any activity which they will audit. Persons having direct responsibility for performance of the activities are not involved in the selection of an audit team.

5.1.3 Audit team members shall have received appropriate indoctrination and training for auditing.

5.2 Audit Planning

5.2.1 The QA Manager. or designee shall develop an audit plan which shall be the basis for the audit. The audit plan is documented on Audit Plan Form (Se Appendix I).

5.2.2 The QA Manager shall develop an audit checklist appropriate to the activity or area being audited. The checklist should contain auditable requirements extracted from the QA Manual,

applicable SOP's or guidance documents. such as EPA SW846. Checklists are designed for each Department by the QA Manager and can be accessed by the QA Department.

5.2.3 The QA Manager shall ensure that the checklist provides an adequate means for indicating whether the question is satisfactorily answered.

5.2.4 Audits are scheduled in a manner to provide coverage and coordination with ongoing QA program activities.

5.2.5 Audits are scheduled at a frequency commensurate with the status and importance of the activity. Within the audit program. each department of the laboratory and each element of the Laucks-QA program is audited. at a minimum. at least once annually.

5.2.6 The QA Manager notifies the audited department. in writing, prior to the audit to provide the subject and scope of the audit. audit schedule. and audit team members. if applicable.

5.3 Audit Performance

5.3.1 The QA Manager and (when required) the appointed audit team members shall proceed through the audit checklist recording evidence of compliance. discrepancies, or recommendations.

5.3.2 During the audit. the QA Manager or appointed team member shall use their best judgment to determine if there is a need to audit at a greater depth than the checklist indicates. If this is the case. the checklist shall be modified accordingly.

5.3.4 Objective evidence is examined. and essential information is recorded, such as the identification of specific evidence examined. specific details of discrepancies or adverse conditions. and applicable references.

5.3.5 The QA Manager shall identify each finding, deficiency, or recommendation in a QA audit report. Findings. deficiencies and recommendations will be listed by department and sequentially numbered in the QA audit report.

5.4 Audit Report

5.4.1 The QA Manager or his designee shall prepare an audit report which should address the following:

5.4.1.1 Date and location (Laucks-department) of the audit.

5.4.1.2 Purpose and scope of the audit.

5.4.1.3 Audit team members (when applicable) and the people contacted during the audit.

5.4.1.4 Description of items. including the rank. type and detail of the audit finding requiring corrective action. The description of the items must be in sufficient detail to enable investigation. evaluation. and correction of the finding. (See Appendix II - Audit Finding Report Form) The report may also include the area affected (See Table in Appendix III) and Finding Type (See Table in Appendix IV)

5.4.1.5 Due date for completion of corrective action plans.

5.4.2 The QA Manager shall issue the audit report to the appropriate levels of Laucks management within four following the audit. This report shall include a copy of each finding. deficiency and/or recommendation.

5.5 Audit Closure and Follow-Up

5.5.1 The appropriate Laucks Management (departmental supervisors, laboratory director hall investigate the reported finding. deficiency or recommendation and do the following:

5.5.1.1 Determine the actions required to correct the discrepancy.

5.5.1.2 Evaluate each discrepancy to determine the root cause of the problem and any generic implications.

5.5.1.3 Determine the corrective action required to correct the discrepancy and to prevent recurrence.

5.5.1.4 Document corrective action and indicate corrective action commitment date.

5.5.1.5 Sign. date. and return the completed form to the QA Manager within the assigned time frame given in the audit report.

5.5.2 The QA Manager shall evaluate each discrepancy/recommendation response. Inadequate or indeterminate responses shall be returned for reexamination of the problem and revised corrective action.

5.5.3 The QA Manager shall verify the corrective action, as stated in the response, and make sure it has been implemented and accomplished as scheduled.

5.5.4 An interim status report of corrective action completion may be issued

5.5.5 After verification of corrective action. the QA Manager shall issue a report stating that all corrective action has been completed and the audit is closed.

5.5.6 If a stalemate is reached concerning either the validity or resolution of an audit finding, affected personnel escalate the concern to the appropriate level of management to effect a resolution.

5.6 Records

The QA Manager shall ensure that the following audit documentation is maintained on file:

5.6.1 Completed audit checklist.

5.6.2 Audit Report (includes findings. deficiencies and recommendations).

5.6.3 Corrective Action (response to discrepancies).

5.6.4 Records pertaining to the completion of corrective action.

5.7 Audit Discrepancy Tracking

5.7.1 Audit discrepancies will be categorized to facilitate tracking and trending of recurrent problems. The categories are as follows:

- Logbook Maintenance
- Document Control Procedures
- QC Procedures
- Standard Operating/Quality Assurance Procedure
- Analytical Method
- Purchasing/Procurement Document Control
- Standards Preparation/Documentation
- Safety/Reagent Labeling or Storage

- Training/Records
- Good Laboratory Practices
- Other

5.7.2 Explanations of Categories Listed Above

5.7.2.1 Logbook maintenance findings include but are not limited to the following: logbooks not being maintained in accordance with Laucks policy, improper entries into logbooks, improper error corrections, logbooks not being kept up to date.

5.7.2.2 Document Control Procedure findings include but are not limited to the following: documents being maintained in such a way that is non-complaint with Laucks document control procedures (this includes archives, SOPs, QAPs, Chemical Hygiene Plan, HTVRs, and forms), records being stored in work areas for longer than 6 months, improper handling of controlled procedures.

5.7.2.3 QC procedure finding include but are not limited to the following: temperatures of ovens and refrigeration units not being monitored in accordance with procedures, balances and pipettes not being verified as required.

5.7.2.4 Standard Operating Procedure and Quality Assurance procedure findings include any case where a procedure has not been followed in full and has not been documented on the applicable corrective action from.

5.7.2.5 Analytical methods findings involve cases where the approved and required analytical method has not been followed to the full extent and there is no documentation that communicates this.

5.7.2.6 Purchasing and procurement document control findings involve instances where the appropriate procedures have not been followed in full. This type of finding includes but is not limited to the following: un-approved use of standards or solvents, lack of certification documentation, etc.

5.7.2.7 Findings for standards preparation and standards documentation include but are not limited to the following circumstances: improperly prepared standards, improperly documented standard preparation, inadequate verification documentation, lack of documentation when procedures are not followed in full.

5.7.2.8 Safety and reagent/chemical labeling findings involve any deviation from approved safety and waste procedures and the chemical hygiene plan.

5.7.2.9 Training and training records findings involve lack of training records, and personnel performing analysis without appropriate qualification documentation.

5.7.2.10 Good Laboratory Practice findings involve significant figures, temperature monitoring, calibration techniques and other associated activities involved with safe and accurate laboratory practices.

6.1 References

Laucks Quality Assurance Plan

Applicable SOPs

Audit Database Tables

Appendix I

Audit Plan Form

Audit Plan

Area to be Audited: _____

Lead Auditor: _____

Audit Team Members (if applicable): _____
_____

Date of Audit: _____

Type of Audit: _____

Checklist(s) to be Used: _____
_____
_____
_____

Individuals Contacted During the Audit: _____
_____
_____
_____

Audit Debrief Date: _____

Report Issued Date: _____

Signature of Lead Auditor: _____

Signature(s) of Team Members: _____
_____
_____

Appendix II

Audit Finding Report Form

Audit Finding Report

Audit Number: Example	Finding Number 1
Facility:	Audit Date:
Auditing Body:	Audit Type:
Lead Auditor:	Affected Area: GC-Semivolatiles

Related Findings:

Finding Rank: Minor Repeat Finding?: No

Finding:

Corrective Action Response:

Opened By:	Date Opened:
Response By:	Response Date:
Corrective Action By:	Scheduled Completion Date:
Verified By:	Date Verified:

Appendix III

Area Names

Depart	Department	DepartmentDescription	Sup ID #
ARCH	Archive	Archive of Documents in QA	0006
BP	Bottle Prep	Bottle Prep	0008
DM	Data Management	Data Management and Administrati	0008
EXT	Extractions	Extractions	0027
GCEF	GC-Extractable Fuels	Extractable Fuels by GC/FID	0038
GCS	GC-Semivolatiles	GC-Semivolatiles	0048
GCV	GC-Volatiles	GC-Volatiles	0038
MSS	GC/MS-Semivolatiles	GCMS-Semivolatile	0048
MSB	GC/MS-Semivolatiles & Volatile	GC/MS-Semivolatiles and Volatiles	
MSV	GC/MS-Volatiles	GCMS-Volatile	0038
SAF	Health and Safety	Health and Safety	0006
HPL	HPLC	HPLC	0038
IN	Inorganics	Metals and Wet Chemistry Office	0053
MIS	LIMS and MIS	LIMs and MIS	0070
MET	Metals	Metals and Metals Prep	0067
MTI	Metals Instrumentation	Metals Instrumentation	0067
MTP	Metals Preparation	Metals Preparation	0067
PM	Project Management	Project Management	0008
QA	Quality Assurance	Quality Assurance	0006
SM	Sales and Marketing	Sales Department	
SC	Sample Control	Sample Control	0008
SP	Special Chemistry	Special Chemistry	0053
WC	Wet Chemistry	Wet Chemistry	0053
YAK	Yakima Office	Yakima Office	0072

Appendix IV

Finding Type

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

Written by: Harry Romberg, Quality Assurance Officer Date: 6-17-96

Approved by: Karen Kotz, Laboratory Director Date: 6/20/96

UNCONTROLLED

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DATA REVIEW SCHEMATIC 10

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

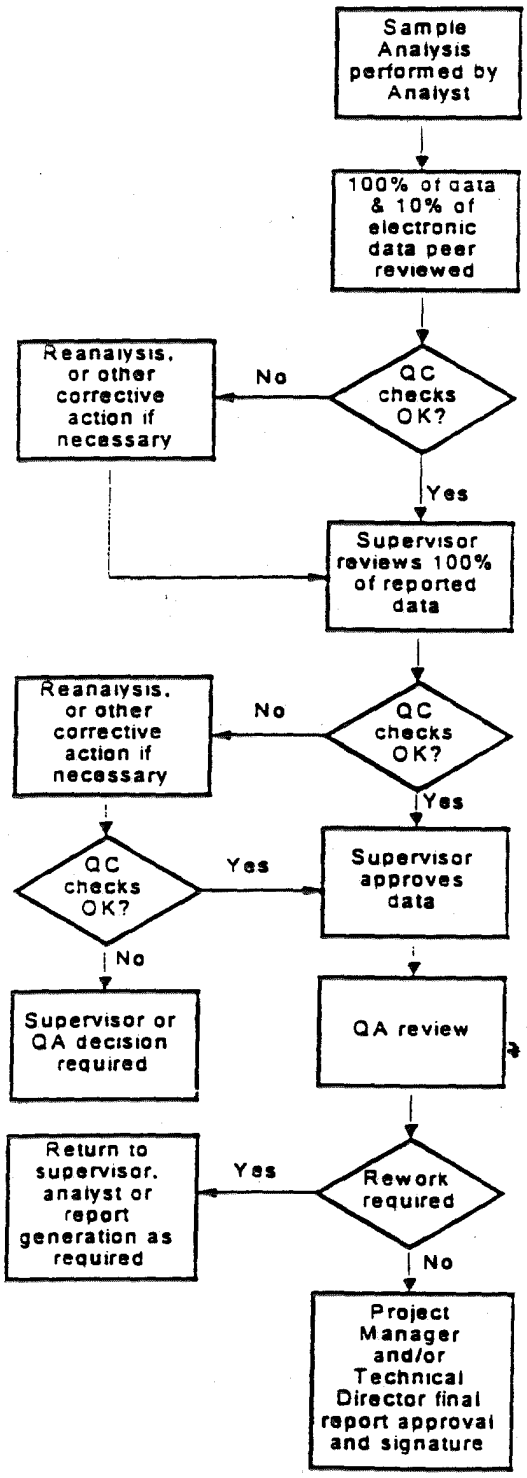
Laucks SOPs

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 % manual entries for transcription errors
-100% of manual calculations for accuracy
-10% spot check of computer calculations
-Check for completeness of raw data or supporting materials
-Confirm spectral assignments and identification of TICs
-Check for appropriate use of significant figures and rounding
-Check for compliance with Method or Project QC requirements
-Check for descriptions of deviations from Method or Project QC requirements
-Check reported values for dilutions
-Check reasonableness of data

-Check for reasonableness of reported data
-Check for completeness of the reported information
-Check for compliance with required QC practices
-Check for descriptions of deviations from Method or Project QC requirements
-Check the information for the report narrative

-Check for compliance with required QC practices
-Check for reasonableness of reported data
-Check for descriptions of deviations from Method or Project QC requirements
-Check for compliance with approved SOPs (periodically)

P:\SOP\QA_SOPS\RVW_CHT1.GFC 6/13/96

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1019

Title: **Controlling, Maintaining, and Monitoring Laboratory Logbooks**

Revision history:

Number	Date
0	4/11/96
1	5/13/98

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

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

Controlled Document

No. 20 Assigned to: Tetra

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1. Introduction and Scope

1.1 Scope

1.1.1 The maintenance of logbooks is essential to monitoring all aspects of laboratory operations including instrument and method performance and in tracking analyses. It is also important to confirming instrument performance at the time of specific analyses and in monitoring ongoing or periodic performance degradation and the steps taken to correct or prevent such occurrences. This document applies to all personnel involved in the preparation, control and use of laboratory notebooks.

1.1.2 More specific instructions for maintaining logbooks can be found in pertinent SOPs, such as LTL-1007 "Maintaining Instrument Records and Logbooks" or LTL-1005 "Analytical Balances" or others specific to other laboratory operations.

1.2 Purpose

1.2.1 The purpose of this SOP is to define the practices used to maintain control and use of laboratory logbooks. This SOP is not intended as a specific description of any particular logbook type but covers the practices that must be in place for all logbooks employed at Laucks.

1.3 Definition of Terms

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

2. Requirements

2.1 Control

2.1.1 Documents that specify quality requirements or activities affecting quality or evidentiary activities shall be controlled to ensure that correct documents are being used and properly archived when completed.

2.2 Maintenance

2.2.1 Quality assurance records (logbooks) shall be compiled and maintained in accordance with approved procedures.

2.3 Monitoring

2.3.1 Logbooks should be periodically monitored to ensure they are being properly maintained and information is being correctly recorded. Standard logbooks and run logs should be monitored at least semiannually by group supervisors or their designees. Maintenance and other logbooks need only be reviewed annually, unless previous review has demonstrated inadequacies in the logbook which require more frequent monitoring.

3. Responsibilities

3.1 Quality Assurance Manager

- Maintain the logs for control of laboratory notebooks and provide control numbers and labels as required.
- Approve format and proposed content of laboratory notebooks; minor changes to pre-printed forms do not need QA approval as long as their basic content does not change.
- Maintain master copies of notebook pages (in instances where pre-printed pages with a specific format are used); this may be in electronic or hardcopy form or both.
- Monitor satisfactory implementation of the requirements of this SOP

3.2 Responsible Supervisor

- Determine the format and content of notebooks used in their respective areas.
- Ensure that QA has been provided with an electronic version of all pre-printed logbook pages in order that they are later available for reprinting or editing.
- Ensure that all laboratory notebooks are properly labeled, including the appropriate control number.
- Ensure that personnel are adequately trained in the proper use of laboratory notebooks
- Periodically review laboratory notebooks to verify satisfactory implementation of the requirements of this SOP. Standard logbooks and run logs should be monitored at least semiannually by group supervisors or their designees. Maintenance and other logbooks need only be reviewed annually, unless previous review has demonstrated inadequacies in the logbook which require more frequent monitoring. This activity may be assigned to another individual but should not be the same individual who regularly completes the log itself.

3.3 Analyst

- Ensure that they are using the appropriate logbook and understand how to properly fill in the required fields.
- Ensure that any new logbook has been given a logbook number by QA before beginning to use it.
- Ensure that the logbook is clearly identified with an instrument ID and purpose or other appropriate title which will enable the analyst to easily identify the logbook.
- Ensure that if pre-printed logbook pages need to be modified, the modifications are approved by their supervisor and that an electronic copy or, if requested an original hardcopy have been provided to QA.

4. Procedure

4.1 Notebook Structure

4.1.1 Laboratory notebooks may be either bound or unbound as described below. Most logbooks should be bound in some fashion but it is recognized that this is not always possible, such as for vendor service records. These records may be stored in 3-ring binders or other suitable notebooks.

4.1.2 In some instances, logbooks may be created from instrument printouts or other pages that do not lend themselves to being pre-bound. In these instances, the log sheets may be stored in a 3-ring binder or other storage until enough sheets have been accumulated to have them bound with the laboratory comb binder.

4.1.3 All logbooks whether bound or unbound must be controlled by QA as designated by the appropriate QA Book Number label (see example in Appendix 1).

4.1.4 Bound notebooks shall conform to the following:

- Where feasible, binding will be of a type that will make the removal and reinsertion of pages readily noticeable.
- If pre-printed and bound, all pages will be sequentially pre-numbered. If the format of the notebook permits the use of the reverse side of the pages, both sides of each page will contain a sequential page number.

- Each page of the pre-printed bound notebook will contain, as a minimum, the laboratory name, logbook title, and sequential page number. Other elements may also be necessary for any specific logbook.

4.1.5 Unbound notebooks shall conform to the following:

- Unbound pages will be contained in a binder or folder that provides protection from damage.
- Each unbound page will contain a unique identifier (e.g., run number/date). For identification purposes, a continuous printout on fanfold computer paper requires only one identifier unless the sheets are separated.
- As noted above in 4.1.2, some unbound logbooks may eventually be bound if practical.

4.1.6 All notebooks will contain the following information on the cover:

- Laboratory name, Laucks Testing Laboratories, Inc.
- Control number assigned by the Quality Assurance Officer
- The department to which the logbook was issued
- The use of the logbook (i.e. balance calibration, instrument run-log, etc.)
- The department book number or title uniquely identifying that book, as required to identify the specific use of the book. This may include an instrument number or other logbook ID (such as a standards logbook ID). This is in addition to the QA logbook number.
- Start Date, the date on which the first entry was made
- End date, the date on which the last entry was made

4.2 Control of Logbooks

4.2.1 The QA Officer will maintain a master log of laboratory notebooks that contains as a minimum, the following information:

- Unique control number for each logbook
- Logbook title, which should reflect the type of information to be entered.

- Department to whom issued, for accountability only. A logbook will generally be assigned to a work station or function, and in no way is a laboratory notebook to be considered a "personal" notebook.

- Date issued, for accountability only.

- Date closed, for accountability.

4.2.2 Master sheets for each logbook will be maintained by the QA Officer and will be utilized for producing notebooks when required.

4.3 Use of Laboratory Logbooks

4.3.1 The notebook is the basic document for recording information. Entries should be made into the notebook in real time, not written on scratch paper and transferred later.

4.3.2 Handwritten entries should be legible and entered in black or blue indelible ink.

4.3.3 Computer-generated data should be printed out and collected at appropriate times to represent the activities being recorded.

- Computer printouts may be either placed in unbound notebooks as described above, or inserted into bound notebooks.

- Computer printouts or other material inserted into bound notebooks must be securely fastened (tape is preferred) in such a way that removal and insertion of material can be determined readily.

4.3.4 When information from related activities is recorded in more than one notebook, provide adequate cross-reference information in all affected notebooks so that all pertinent data can be readily accessed.

4.3.5 Do not skip pages when entering data. For example, if data is not readily available for entry, do not leave space for later entry. Enter the data when it becomes available and provide adequate cross-references if required.

4.3.6 In cases where partial or complete pages must be left blank and not used, indicate the unused portion by placing a horizontal line at the beginning and end of the unused portion and connecting opposite ends of the horizontal lines with a diagonal, resulting in a Z-shaped figure. The individual striking out the blank area will initial and date the diagonal.

4.3.7 Errors or other changes must be deleted in a similar fashion or with a single-line cross-out which has been initialed and dated. **No erasures, overwriting, white-out or multiple-line cross-outs (blacking out) are acceptable.**

4.3.8 When pre-printed formats are used and all possible entries are not required, the remaining blanks may be struck out with a Z as described above, or entries such as N/A may be placed in the unused blanks.

4.3.9 The individual entering information into the notebook shall initial and date each page used, or in the case of logbooks with ongoing records which do not occupy the entire page, such as maintenance logs or balance logs, each individual entry.

4.4 Supervisory Monitoring of Laboratory Logbooks

4.4.1 Standard logbooks and run logs should be monitored at least semiannually by group supervisors or their designees. Maintenance and other logbooks need only be reviewed annually, unless previous review has demonstrated inadequacies in the logbook which require more frequent monitoring. This activity may be assigned to another individual but should not be the same individual who regularly completes the log itself.

4.4.2 Logbooks should be reviewed using the review items provided in Appendix II, although it is not necessary to actually document the review using this checklist.

4.4.3 Errors should be formally brought to the attention of the responsible individual through the use of Corrective Action Forms. If errors are correctable or items can be corrected for legibility problems, they should be corrected using the proper error correction technique.

4.4.4 Logbooks that have been reviewed are marked with a fluorescent yellow or other colorful label that looks similar to the label in Appendix III.

4.5 QA Monitoring of Laboratory Logbooks

4.5.1 The QA Officer will verify during periodic audit and surveillance activities that notebooks are properly completed and maintained. This will generally be done approximately annually as part of routine audits. This observation does not preclude the requirement for supervisory review.

Appendix I

Example QA Logbook Label

Laucks
Testing Laboratories, Inc.

QA Book No.: _____

Issued To: _____

Used For: _____

Dept. Book No.: _____

Start Date: _____

End Date: _____

Appendix II

Example Logbook Review Items to be Observed

- Have all pertinent fields been filled or marked not applicable (N/A)?
- Has empty space been crossed out properly initialed and dated?
- Have errors been corrected with single-line crossouts, initialed and dated (no obliterations or overwrites)?
- Are all entries clear and easy to read and comprehend?
- If calculations are involved, check several random calculations for error.
- If traceability is involved (as for standards) check several random entries to confirm that the logbook entries can be tracked back to the original entry.
- If standards log, observe some actual standards and compare them against logbook entries for accuracy.
- Are all handwritten entries initialed and dated?
- If the book is beginning to deteriorate, it should be repaired or retired and replaced.

Appendix III

Example Logbook Review Label

Logbook pages _____through_____
have been reviewed for completeness and
spot-checked for accuracy>

Initials:_____ Date:_____

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-2001

Title: **Waste Segregation and Disposal**

Revision history:

Number	Date
1	4/10/91
2	9/11/91
3	4/26/94
4	4/09/97
5	5/12/98

Written by: Harry Romberg Date: 6-2-98
Harry Romberg, Quality Assurance Officer

Approved by: Kathy Kreps Date: 6-2-98
Kathy Kreps, Laboratory Director

Controlled Document

No. 20 **Assigned to:** Tetra

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HAZARDOUS WASTE STICKER 10

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CORROSIVE AND FLAMMABLE STICKERS 11

1. Introduction and Scope

1.1 Method Description

1.1.1 The purpose of this SOP is to describe the laboratory waste disposal scheme currently in place at Laucks. The primary waste streams described include solvents, PCB oil wastes, COD and TOC waste and soil samples. This SOP only covers handling of the waste from the point of collection.

1.1.2 This method is restricted to use by, or under the supervision of analysts experienced in the techniques described. As part of their training for analytical tasks which generate related wastes, each analyst must be trained to properly dispose of the waste or to the consolidate it at the appropriate collection point.

1.1.3 This SOP generally does not cover handling of the waste up to the point of disposal.

2. Equipment List

2.1 Equipment

2.1.1 The equipment necessary to properly dispose of laboratory wastes varies with the type of waste. In general, an appropriate container, packing material, and safety equipment (including clothing, eye wear, and respirators) is required.

3. Safety precautions

3.1 Safety Precautions

3.1.1 Solvent wastes may contain materials flammable at room temperature or lower. Caution should be taken to avoid flames and sparks when in the presence of or handling these wastes.

3.1.2 COD and TOC wastes may contain materials which will burn the skin, eyes, and/or mucous membranes if improperly handled. Precautions should be taken to avoid accidental contact.

3.1.3 All wastes may contain materials which can have both known and unknown long-term health effects. COD and TOC wastes, for instance, contain high levels of mercury salts. Direct contact should be avoided through the use of proper clothing and eye wear, even if no immediate danger is obvious. In the case of volatile solvents and other materials, handling should be done in a well-ventilated area and the exposure to vapors minimized. Where strong fumes are unavoidable, a carbon-filter or other respirator should be worn.

3.1.4 All people who handle waste products or the original reagents should be aware that the laboratory provides safety equipment and has a file containing Material Safety Data Sheets (MSDSs) on all laboratory chemicals in support of OSHA and other safety programs.

4. Operation procedures

4.1 Operations Appropriate to All Collection Areas

4.1.1 Land Disposal Restriction Forms (LDRs), manifests and other paperwork are not extensively discussed in this SOP because the disposal vendor deals with this aspect of the paperwork. It will only be necessary for the person who will be asked by the vendor to sign these forms (usually QA) to check that the information on the forms is accurate and to sign the form.

4.1.2 All waste requiring a Hazardous Waste disposal sticker and manifest will be marked with one of two EPA Hazardous Waste Site numbers. All waste transported from the 921 facility will be numbered WAD981762024 and all waste transported from the 940 facility will be numbered WAD027446608.

4.1.3 The Hazardous Waste Sticker must be labeled with the proper DOT shipping name, even though the disposal company will usually replace the label before shipping. The proper shipping names are listed below in the applicable sections of this SOP.

4.1.4 All collection drums **must** be marked with an appropriately filled out Hazardous Waste sticker (see Appendix A). It is only necessary for Laucks staff to fill in the date that collection was **started** and the contents of the drum in the appropriate space. Hazardous Waste cannot be accumulated for longer than 90 days before it must be disposed. Therefore, do **not** mark the date on the drum until collection is started so as to maximize the allowable time until disposal. This sticker will be replaced by the transporter when they arrive to transport the waste to an approved disposal facility. The replacement sticker will contain all of the information required for transport and disposal.

4.1.5 In addition, corrosive and flammable waste collection drums must have a sticker which indicates their corrosive or flammable nature (see Appendix B).

4.1.6 Once a material has been designated as waste and disposed into the designated drum, that drum must not be stored for longer than 90 days from the point that collection was **started**. This is rarely of concern at Laucks because transport is generally scheduled for most wastes within much less time than the required maximum storage time.

4.1.6.1 The one variation from the above rule is the TOC waste drum. This drum is not a satellite collection point but is actually the catch drum for the waste directly from the instrument. It will be disposed as soon as possible after it has reached capacity.

4.1.7 When collection drums are full or the 90-day limit is approaching, the Quality Assurance (QA) Department must be notified. The preferred lead-time for pickup is 10 working days so QA should actually be contacted 80 days after collection is

initiated. This department, at the time of this writing, is responsible for contacting the appropriate approved transporter and insuring proper disposal takes place.

4.1.8 All questions or concerns regarding hazardous waste operations should first be directed to QA who will determine the appropriate course of action.

4.2 Mixed Solvent Waste

4.2.1 This waste stream is primarily composed of methylene chloride with some acetone and hexane and potentially small quantities of other solvents or dissolved products. The collection point for all of this waste is in the 921 facility (Extractions) solvent locker.

4.2.2 Small, 5 gallon or less containers of other mixed solvent waste may be collected as satellite accumulation units in the inorganics or organics instrument preparation areas but these **must** be transported to the primary drums in Extractions when full. Satellite accumulation containers must be kept closed when not in use and must be marked with the words "Hazardous Waste" or with other words that identify the contents of the container. This will most conveniently be done by using a blank Hazardous Waste Sticker.

4.2.3 When new materials are collected in the primary drum, a Hazardous Waste sticker should be affixed with an initial collection date. The Hazardous Waste stickers should be marked with a DOT shipping name of **"Waste Flammable Liquids"**.

4.2.4 Although methylene chloride is non-flammable, other components of these waste drums may be highly flammable. Thus, all of the waste solvent containers must be labeled as flammable.

4.2.5 At least 2 inches of headspace must be left between the top of the liquid and the top of the drum to allow for expansion.

4.2.6 When 3 or more full 55 gallon drums of this waste have been accumulated or 80 days have passed since the beginning of collection of the oldest accumulated drum, QA must be contacted to arrange for transport and disposal.

4.2.7 At the time of this writing, Laucks uses Laidlaw Environmental as the facility of choice for handling this waste stream, although this could be changed at the discretion of QA on either a one-time or ongoing basis.

4.3 Chemical Oxygen Demand (COD) Waste

4.3.1 The primary constituents of this waste are sulfuric acid, water, mercury, silver, and chromium (both tri- and hexavalent). The collection point for this waste is in the inorganics area where CODs are analyzed. These analyses are conducted in small pre-packaged tubes. The reacted tubes are not considered to be waste until they are poured out of the tubes into a collection container.

4.3.2 Collection containers must be labeled with a Hazardous Waste sticker as previously noted. The Hazardous Waste Sticker should be marked with a DOT shipping name of **"Waste Corrosive Liquids, Acidic, Inorganic"**.

4.3.3 In addition to the hazardous waste sticker, these containers should be labeled as corrosive with the appropriate sticker as previously noted.

4.3.4 The waste not held for more than 90 days from initial collection (after pouring from the reaction tubes) until transportation for disposal. After 80 days have passed since the beginning of collection, QA must be contacted to arrange for transport and disposal within the allowable timeframe.

4.3.5 At least 2 inches of headspace must be left between the top of the liquid and the top of the drum to allow for expansion.

4.3.6 At the time of this writing, Laucks uses Laidlaw Environmental as the facility of choice for handling this waste stream. This vendor can be changed at the discretion of QA on either a one-time or ongoing basis.

4.4 Total Organic Carbon (TOC) Waste

4.4.1 The primary constituents of this waste are mercury, potassium persulfate, nitric acid, and water. This waste is collected directly from the instrument into a waste container beneath the instrument.

4.4.2 As this is a continuous process, Laucks does not begin the 90 day clock before disposal is required until this container is full. However, the container must be marked with a corrosive sticker. The Hazardous Waste sticker, in this case, must be dated as soon as the container is full and affixed at that time. The Hazardous Waste Sticker should be marked with a DOT shipping name of **"Waste Corrosive Liquids, Acidic, Inorganic"**.

4.4.3 At least 2 inches of headspace must be left between the top of the liquid and the top of the container to allow for expansion.

4.4.4 As soon as the container is full, the QA department must be notified to arrange for disposal. This waste stream will generally not be held in storage for very long after collection.

4.4.5 At the time of this writing, Laucks uses Laidlaw Environmental as the vendor of choice for handling this waste stream. This vendor can be changed at the discretion of QA on either a one-time or ongoing basis.

4.5 Soil Sample Disposal

4.5.1 State law allows a laboratory to store samples and other materials indefinitely, until they are considered waste and disposed. After that time, from the date of first accumulation, a 90 day timeframe is allowed before disposal must occur. Thus, soils

should not be disposed of until enough have been accumulated to fill at least one 55 gal. drum.

4.5.2 Each drum used for soil waste disposal must be clearly marked with an identifying number which will be used to track which drum contained which samples. When samples are signed-out from their storage areas for disposal, the log sheet must be appropriate marked with the assigned drum number. This will enable the laboratory to track which samples were disposed in which drum.

4.5.2.1 The drums should be marked with a year, location from which they originate, and sequential number. Thus drums for which accumulation began in 1998 from the extractions laboratory would be marked 98-921-01. The -01 being a sequential number that would be incremented with each additional drum -02, -03, etc. throughout 1998. A drum from the main lab would be designated 98-940-01, etc.

4.5.2.2 When samples are transferred from the storage locations to the drums, the Secure Storage Custody Log must be marked to indicate into which drum they were disposed. This should include any bottle identifiers, if necessary to identify just what was disposed. Thus, it will be necessary for personnel disposing of samples to check the drums to make sure there is enough room for the designated samples. Soil samples will generally have their lids removed and disposed in the regular garbage. The jar and all will then be disposed in the waste drum. If the lids themselves contain client identifying marks or locations or have significant amounts of adhering material (oil, etc.) which cannot be readily dumped into the drum the lid will also be disposed into the waste drum.

4.5.2.3 When the drums are disposed, it will be necessary for the laboratory representative who signs the manifest to mark the drum identity on the manifest, although this only needs to be on the laboratory copy if the transporter does not want this information to appear on their copy of the record.

4.5.3 QA must be notified 80 days after accumulation has begun in order to arrange for disposal in a timely manner. If samples are not disposed until there is enough to fill a drum, this timeframe is not of major concern because there are always Hazardous Waste pickups scheduled within any 90 day time period.

4.5.4 The only stickers these drums must have is the Hazardous Waste sticker with the date accumulation was started clearly marked. The Hazardous Waste Sticker should be marked with a DOT shipping name of "**Waste Environmentally Hazardous Substances**".

4.5.5 At the time of this writing, Laucks uses Laidlaw Environmental as the vendor of choice for handling this waste stream, although this could be changed at the discretion of QA on either a one-time or ongoing basis. This vendor incinerates these soils prior

to landfilling which should dispose of any organic materials, including labels, oily material and other hazardous organic substances.

4.6 PCB Oil Waste Disposal

4.6.1 Laucks no longer analyzes many oil samples for PCBs. Thus, this is a very small and infrequent waste stream. However, discussion is presented here in order that there be some documented course of action when it is necessary to dispose of these materials.

4.6.2 All oil samples which are analyzed for PCBs or otherwise known to contain PCBs are treated as PCB oils. No effort is made to distinguish those that actually do contain PCBs.

4.6.3 These oils are accumulated in a 5 gal. drum located in the Extractions laboratory warehouse. This metal drum is stored inside of the lower half of a cut-off plastic 55 gal. drum which fulfills the federal requirements for secondary containment during storage.

4.6.4 When a full drum has been accumulated, Eastern Electric is contacted for pickup and disposal. A signed receipt must be obtained as proof of disposal. Eastern Electric sends a manifest in subsequent mail within 35 days of waste pick-up and must also send a certificate of disposal within 30 days after the actual disposal date.

4.6.5 No annual report to the Department of Ecology is required because the level of PCBs is considered so high as to fall outside of the state's responsibility to monitor. At such levels the federal government regulates the disposal under TSCA. For this reason, it also does not fall within the federal requirement for RCRA governed waste disposal within 90 days. Eastern Electric is responsible for filing appropriate reports. TSCA regulations require that manifests and certificates of disposal be kept on file for a minimum of 3 years.

5. Reports

5.1 Disposal Paperwork

5.1.1 Our current vendor produce all of the required paperwork and insure all of the appropriate container markings (stickers, etc.) are in place prior to shipment. Since Laucks' waste streams are consistent from time to time, our vendors already have the information required to properly fill out the paperwork and Hazardous Waste stickers.

5.1.1.1 The paperwork includes the manifests, land disposal restriction forms and other shipping paperwork. Thus the only requirements of the laboratory are to insure the paperwork is accurate and to sign the appropriate forms.

5.1.2 After the waste has been transported to the disposal or accumulation facility, a signed manifest is returned to the laboratory. This is kept with the permanent record.

5.1.3 All certificates of disposal later provided by the disposal vendor are also associated with any waste shipment and kept with the permanent record.

5.1.4 All records are retained for at least 5 years from the date of shipment of the waste.

5.2 Annual Reporting Requirements

5.2.1 The laboratory must file an annual report with the Washington Department of Ecology (WDOE) for legal and tax purposes. This report is due on March 1 each year. Reports are filed for both the 940 and the 921 facilities (both EPA ID numbers). All waste transported from the 921 facility will be numbered WAD981762024 and all waste transported from the 940 facility will be numbered WAD027446608.

5.2.1.1 The only exception to this reporting requirement is the reporting of the PCB waste oil which is a federally regulated waste and is thus not reported to the WDOE.

5.2.2 The format of this report is defined by WDOE in books provided to the laboratory several months in advance of the due date. Details of this report are not provided in this SOP.

5.2.3 In addition, as part of a WDOE program to reduce hazardous waste in general, Laucks files an annual pollution prevention plan update in September of each year. This report is more loosely defined and the only major requirement is that it be filed. Details of this report are not part of this SOP.

Appendix I

Hazardous Waste Sticker

See directions in this SOP for proper filling out of this sticker.

HAZARDOUS WASTE

FEDERAL LAW PROHIBITS IMPROPER DISPOSAL

IF FOUND, CONTACT THE NEAREST POLICE, OR
PUBLIC SAFETY AUTHORITY, OR THE
U.S. ENVIRONMENTAL PROTECTION AGENCY

PROPER D.O.T.
SHIPPING NAME_____UN OR NA#_____

GENERATOR INFORMATION:

NAME_____

ADDRESS_____

CITY_____STATE_____ZIP_____

EPA
ID NO._____

EPA
WASTE NO._____

ACCUMULATION
START DATE_____

MANIFEST
DOCUMENT NO._____

HANDLE WITH CARE!

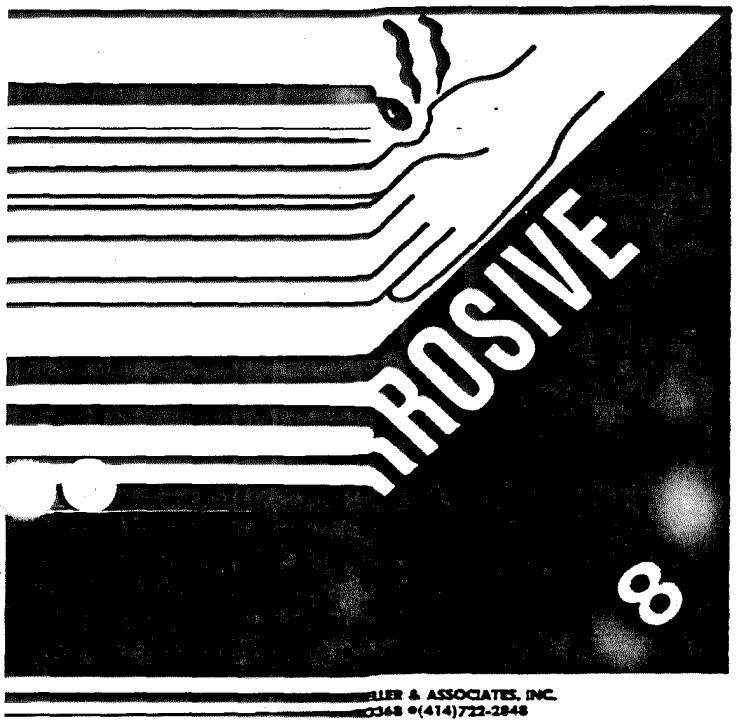
CONTAINS HAZARDOUS OR TOXIC WASTES

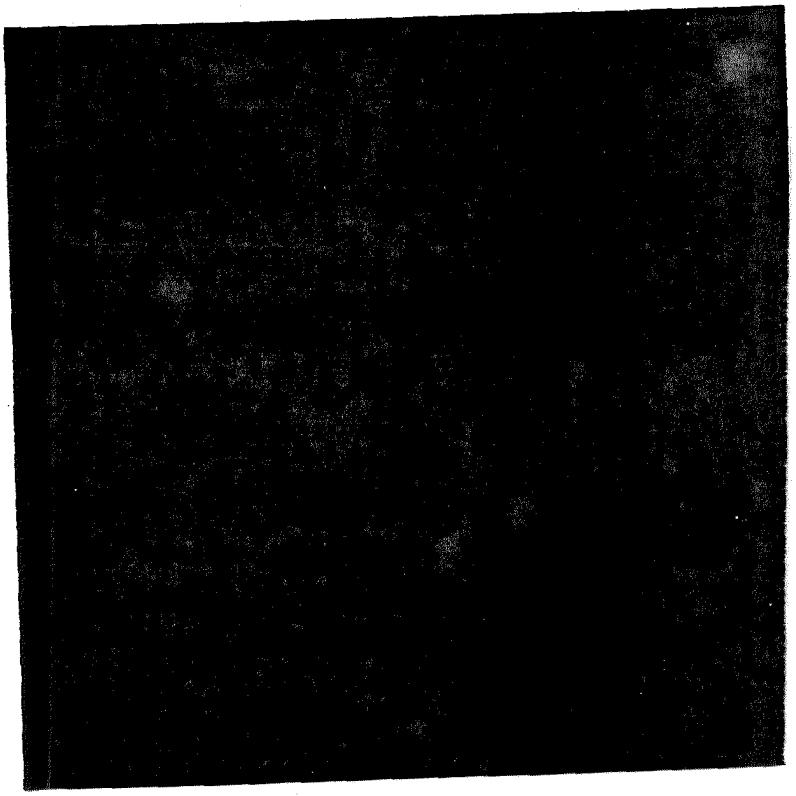
STYLE WM-6

Printed by LABELMASTER, Div. of AMERICAN LABELMARK CO., INC., CHICAGO, IL 60646

Appendix II

Corrosive and Flammable Stickers





LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-4002

Title: **Electronic Sample Entry and Log-In**

Revision history:

Number	Date
5	06/19/96
4	12/28/95
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2	06/05/90
1	04/24/90

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
9401493                                                                  SAMPS
 KEYS    ST  ORDER # 94-01-496                          RECEIVED 01/12/94   2
9401485 WR                                              DATE DUE 01/26/94
9401486 TR   CLIENT C.H. NIECE        INVOICE BY R      VER BY
9401487 TR  PROJECT                   % DISCOUNT        KEEP FOR 45
9401488 WR  CONTACT KARIS            TEST/JOB           KEEP TIL 03/12/94   DP/TST
9401489 TR      CAT 1436759          % SURCHARGE            DISP D
9401490 TR                          #REP/INV 3  3
9401491 WR                                              QUOTED $            MS   4
9401492 WR  COMPANY C.H. Niece & Co.                    SAMPLE $ UNKNOWN
9401493 TR    FACIL Vashon, WA 98070                      MISC $
9401494 WR                                               TOTAL $ UNKNOWN
9401495 TR      REP Larry Niece                            INV #
9401496 WR    PHONE (206)463-5281
9401497 WR                                              CREATED 01/13/94
9401498 TR  WORK ID Wesleyan Community Church           WRITTEN 01/13/94
9401499 WR    TAKEN Client                             TRANSMIT
9401500 WR    TRANS UPS                                COMPLETE
9401501 WR     TYPE Water                              REPORTED
9401502 WR    ATTEN Larry Niece                        INVOICED
              P.O. #                                WRITTEN BY PAMJ
                                                                   CL NO 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
9404[illegible]
  KEYS                                                  DEPTS/TESTS
9404176-12A   ORDER# 94-04-191/WR CON MARKR                                HIST1
9404176-13A   CLIENT PETER PAN    CAT                                      HIST2
9404176-13B  WRITTEN 04/05/94      BY HOLLYW
9404176-13C    DASH # 02 A CATEGORY
9404176-13D  SAMPLE ID Mahi Mahi Lot #X3159 (#2)
9404176-14A  COLLECTED 04/05/94            QA/QC   MAT E
9404176-15A     STORED Analyst/Freezer   RECVD 04/05/94 SC   2
9404176-15B                               DUE 04/15/94
9404176-15C  JOB HIST2  FL   QC?         QUOTE    75.00
9404176-15D NAME Histamine                SDG#          StID#
9404176-16A TESTS:DEP/CAT/NAME/D/R
9404176-17A    HISTAM
9404176-17B
9404176-17C
9404176-17D
9404176-18A -SAMPLE AND WORK DESCRIPTION-
9404191-01A  This job code is for any additional samples
9404191-02A
                                                                  CL NL SL J1
F5=Result,Price  F6=Lookup,NextJob,StateID   F10=More   Shft-Fn=Fn Help
```

FRAC Screen

4.2.5. All sample bottles are numbered with the work order number and a fraction (or sample) number. Fraction numbers are assigned sequentially to each sample based on the order in which the samples were sorted and logged (COC order, client sample number order, etc.). This number is used to track the sample throughout the laboratory. See section 4.2.10 for specifications for unique bottle identifiers required by Navy projects.

4.2.6. A sample can be uniquely identified by its work order number and the fraction number. For instance, if the work order number was 9004001 and there were 4 samples, the job would consist of samples

9004001-01
9004001-02
9004001-03
9004001-04

4.2.7. Each sample might consist of several containers, all of which would be marked with the same sample number.

4.2.8. If necessary, more than one fraction may be created for a sample (generally, this is related to billing issues - when one analysis is discounted in price and another is not, for instance), but they will all bear the same fraction number and be differentiated by an automatically-assigned letter suffix. For instance, if sample 9004001-01 had 3 fractions, purely for internal accounting reasons, the three fractions would be identified as:

9004001-01A
9004001-01B
9004001-01C

4.2.9. The person performing log-in needs to be aware of this effect, but it has no impact on sample identification within the lab, on sample tracking, or on the sample number placed on the bottles/jars. In the above example, all bottles submitted for this sample would be marked 9004001-01.

4.2.10. For Navy projects, each bottle must have a unique bottle identifier. Every bottle must have a specific 1-3 digit numerical identifier that is unique to each bottle submitted within a workorder. The numbers are assigned in consecutive order so that all bottles of similar size/type with the same preservation for the same analysis (analyses) from a particular workorder will have consecutive bottle numbers. The first bottle of each analysis type in each new workorder starts over again with bottle number 1.

4.2.11. This information must be recorded in the "Bottles" computer tracking program under each workorder and the workorder-unique bottle identifier will be printed in the bottom left corner of each bottle label (which also contains the workorder number and the sample number) before the bottle label is affixed to each individual bottle.

4.2.12. ALERT: Each SAM work order can accommodate up to 57 fractions and no more. Each work order must allow sufficient fraction space for later changes or additions. Therefore, no more than 50 samples should be logged into any single work order. If, for administrative reasons, some or all of the samples consist of more than one fraction, then no more than 50 fractions can be logged. Should the submittal consist of more than 50 samples, or more than 50 fractions, initiate additional work orders as required. Cross-reference the work order numbers, so that all samples submitted together can be reported together to the client. You can perform this cross-reference manually (by noting on accompanying documents "See Also [Work Order Number]) or you can make appropriate comments in the Work Order Comment field (F2). To the degree possible, make sure that multiple work orders which represent one complete project in the client's mind are created sequentially, with no other unrelated work order numbers intervening.

4.2.13. Additionally, Sample Delivery Groups (SDGs) are commonly created for project work at the time of sample entry. SDGs consist of no more than 20 samples being analyzed for the **same** test. This is in order that the appropriate amount of QC may be analyzed and reported with any sample set. Specifics of the SDG creation process are outlined elsewhere in this SOP.

4.2.14. ALERT: Each fraction will accommodate only 27 tests. If more than 27 analyses are required on any sample, additional fractions should be made (ie -1A, -1B, -1C, etc.).

4.2.15. ALERT: The work order will accommodate only one date of receipt, while each fraction will accommodate individual receipt dates and due dates. If samples are submitted over several days, and are logged into one work order, the Sample Custodian **MUST** enter appropriate dates of receipt in each fraction. The FRAC program will default to the current date. If the samples were received on an earlier date, that date **MUST** be entered for that fraction for the date to be correct. Similarly, the fraction due date will default to that of the workorder on the ORD screen. If different fractions of the same sample are due at different times, due to client or other demands, the date they are due **MUST** be entered for that fraction.

4.3. Special documentation procedures for CLP samples

4.3.1. Completion of the CLP Sample Receipt Log, and the Supplemental Sample Receipt Log

4.3.1.1. CLP Sample Receipt Log and the Supplemental Sample Receipt Log are CLP-specific sample login sheets. For each cooler received a CLP Sample Receipt Log Form and a Supplemental Sample Receipt Log must be completed. This form takes the place of the In-House Sample Receipt Log (Appendix 2). Copies of these forms may be found in Appendix 3.

4.3.1.2. Complete the header information requested at the top of the forms. Use multiple pages if necessary.

- date received
- time received
- client name
- SDG #
- COC # (if available)
- sample log-in date
- work order #
- client project
- airbill number (if available)
- and initials of the person logging in the samples.

4.3.1.3. Complete the Non-Conformance check list. If there is a problem with the custody seals, chain of custody records, or agreement between the custody records, the client must be contacted. In this case, this should be done **in writing** (preferably FAX) by the appropriate project manager. A copy of the communication must be kept with the COC in the work order file.

4.3.1.3.1. Since the extractable fractions will be transferred to the extractions lab, a Secure Storage Custody Log must be completed, and the samples are held on 8C in the WO1 walk-in cooler (extractions hold shelf) pending pick-up by extractions personnel. Specifics of the Storage Custody Log is outlined in the Chain-Of-Custody SOP located in the SOP manual.

4.3.2. Assignment of SDG numbers

4.3.2.1. The SDG name is assigned by sample control and is usually based on client name or project name followed by sequential numbering.

4.3.3. Assignment of lab quality control samples

4.3.3.1. The client may choose to designate which samples are to be analyzed as matrix spike/matrix spike duplicate samples. This means that the sample preparations and the VOA departments cannot self-assign QC samples until all samples from the SDG are received. It is the responsibility of the sample login person to notify the operations staff when a specified QC sample is received.

4.3.3.1. Note in the **SAM SDG** records which sample is QC-assigned. This information is pulled into SDG from the FRAC program when the SDG is created.

4.3.3.2. On the FRACTION screen, an X is placed in the QC field. This will print out next to the sample and indicate that it is an assigned QC sample.

4.3.3.3. Writing a department comments message for whichever department (EX, MS, and/or GC) should be notified.

4.3.4. Completion of SDG records.

4.3.4.1. For each project (NFESC, HAZWRAP, or Army Corps) SDG records in SAM are created. The SDG concept follows the CLP model: that is, up to 20 samples of similar

matrix and analytical fraction are grouped together for preparation and analysis. Samples are assigned to SDGs at sample login and are also reported by SDG.

4.3.4.2. The **SDG** program is an electronic means of compiling information about the samples assigned to an SDG in one place. Example **SDG** screens are reproduced on the following page:

```
                                   SDG Database
CDM      SDG Group : CDM10      Date Due: 03/13/93        Created:  02/19/93
  KEYS   Fraction  : E SPUMITO                           Updated:  02/19/93
BOG05G   Project   : CDM (EDB)              Client:
BOG05I   SAS Number:            Case Number:            Max. Samps:
BOG06G
BOG06I    Work  Samp                                                Associated
BOG07G    Ord   Num  QC            Client ID          Mat TS pH      Blank
BOG07I
BRPTSS
CANONS   9302598 17   07-MW-005-0-000-F                W
CB2SFV   9302598 20   07-MW-007-0-000-F                W
CB3-3V
CBSFXS
CDM10E
CDM10G
CDM10I
CDM10P
CDM10S
CDM10V
CDM11E
CDM11G                                                                 CL NL SL J1
5=Lookup,Copy,Print  6=Toggle     F10=More    Shft-Fn=Fn Help
```

SDG, Screen 1

```
                                   SDG Database
COM        SDG Group : COM10        Date Due: 03/13/93        Created:  02/19/93
  KEYS     Fraction  : E SPVMITO                              Updated:  02/19/93
BOG05G     Project   : COM (EDB)                 Client:
BOG05I     SAS Number:              Case Number:          Max. Samps:
BOG06G
BOG06I      Work  Samp            Date
BOG07G      Ord   Num   VTSR   Collected
BOG07I
BRPTSS
CANONS     9302598 17 02/16/93 02/13/93
CB2SFV     9302598 20 02/16/93 02/13/93
CB3-3V
CBSFXS
COM10E
COM10G
COM10I
COM10P
COM10S
COM10V
COM11E
COM11G                                                          CL NU SL J1
5=Lookup,Copy,Print  6=Toggle     F10=More   Shft-Fn=Fn Help
```

SDG, Screen 2

```
                                   SDG Database
COM        SDG Group : COM10        Date Due: 03/13/93        Created:  02/19/93
  KEYS     Fraction  : E SPVMITO                              Updated:  02/19/93
BOG05G     Project   : COM (EDB)                 Client:
BOG05I     SAS Number:              Case Number:          Max. Samps:
BOG06G
BOG06I      Work  Samp                        Fractions
BOG07G      Ord   Num   EDB
BOG07I
BRPTSS
CANONS     9302598 17   X
CB2SFV     9302598 20   X
CB3-3V
CBSFXS
COM10E
COM10G
COM10I
COM10P
COM10S
COM10V
COM11E
COM11G                                                          CL NL SL J1
5=Lookup,Copy,Print  6=Toggle     F10=More   Shft-Fn=Fn Help
```

SDG, Screen 3

```
                                   SDG Database
CDM                SDG Group : CDM10          Date Due: 03/13/93          Created:  02/19/93
 KEYS              Fraction  : E SPUMITO                                  Updated:  02/19/93
BOGD5G             Project   : CDM (EDB)                  Client:
BOGD5I             SAS Number:                Case Number:                Max. Samps:
BOGD6G
BOGD6I
BOGD7G
BOGD7I
BRPTSS
CANONS
CB2SFU
CB3-3U             Comments:
CBSFXS
CDM10E
CDM10G
CDM10I
CDM10P
CDM10S
CDM10U
CDM11E
CDM11G
                                                                    CL NC SL J1
5=Lookup,Copy,Print  6=Toggle      F10=More    Shft-Fn=Fn Help
```

SDG, Screen 4

4.3.4.3. Fill in the header section of the first screen page. For CLP cases, fill in the Fraction (V=Volatiles, P=Pesticides, S=Semivolatiles etc..) Indicate the project name, and the client's name.

4.3.4.4. When the work order number and sample number are entered, the sample-specific information shown in screens 1, 2, and 3 is read in from the SAM database. (**Hint**: after the first work order number is entered, it is only necessary to enter sample numbers for subsequent samples from the same work order.)

4.3.4.5. On screen 3, a table of fractions/tests is created. An 'X' is entered to signify that a particular test is required on a given sample.

4.3.4.6. Each 'fraction' has a separate SDG entry. For instance, VOAs and ABNs are entered on separate SDG records, as indicated above (V=Volatiles, S=Semivolatiles, P=Pesticides). A single letter suffix (V, S, P etc...) is assigned to each SDG record before it is saved to disk. The end result is that you may have multiple SDG records for a given SDG, each with the same root name, but a different suffix. This system is used to allow for the possibility that within the same SDG, varying numbers of tests will be assigned to samples within that SDG.

4.3.4.7. The last screen page is used for any comments which the sample login person or project manager would like to record for the operations staff.

4.4. Sample storage

4.4.1. The following tests must be started very soon after receipt when performed on water samples.

Test Type/Name	SAM Code(s)
NO_3 - nitrate	NO3ICW
NO_2 - nitrite	NO2_W, NO2_DW
ortho phosphate and soluble reactive phosphate	P04O_W, P04S_W
Cr^{+6} - hexavalent chromium	CR6_W, CR6_WM
CO_2 - carbon dioxide	CO2_N
DO - dissolved oxygen	DO_W
BOD - Biological Oxygen Demand	BOD_5
Chlor A - Chlorophyll A	CHLORA
Settleable Solids	SETSOL, SETSL2
Filtration for dissolved metals	FILTER
pH	PH_EPW, PH_SWW
Microbiological tests	[various]
Color	COL_DW
Turbidity	TUR_TW, TUR_W
Sulfite	SO3_W
MBAS - Methylene Blue Active Substances	MBAS
Chlorine	CL2_R

4.4.2. A rush backlog report is printed throughout the day for short holding-time tests, with the exception of microbiology, in order that they be recognized by the analysts.

4.4.3. For microbiological samples and for samples which arrive late in the day and for which the holding time will expire if the analysis is not started that day, the containers must be taken immediately to the work areas in which the tests will be performed and the primary person responsible for these tests notified that samples are here. A list with the name of the appropriate analyst is posted in the sample entering area.

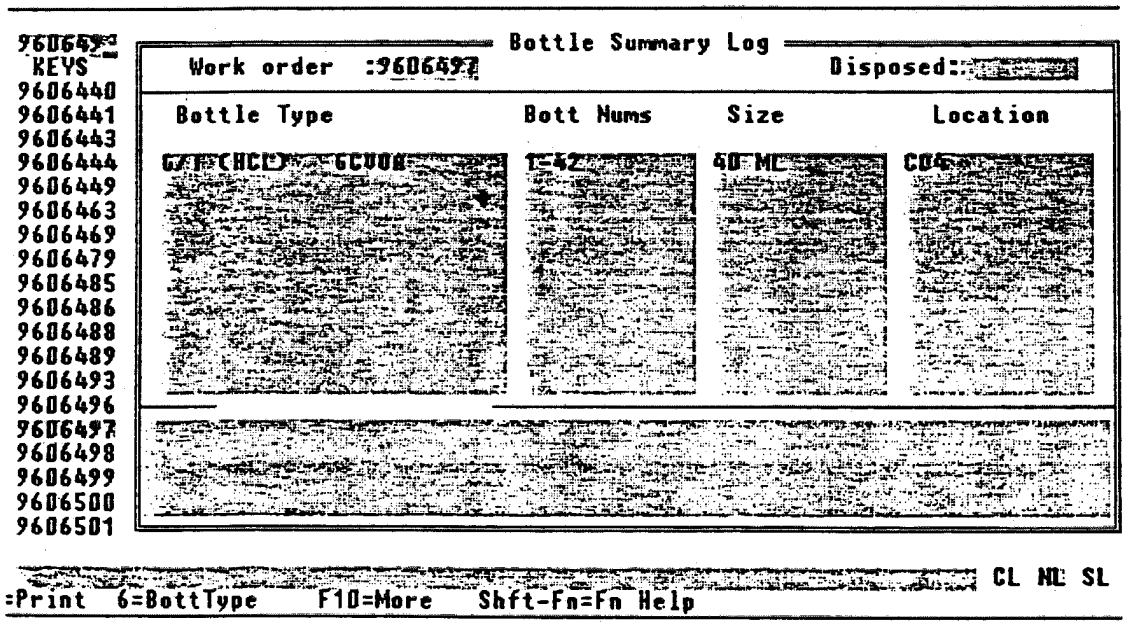
4.4.4. Storage locations

Location	Description of contents
VOA refrigerator	Soil and water volatiles
Shelf	Aqueous metals, oils, no cooling required
Inorganics cooler	Complete small water jobs
Walk-in cooler	All other soil and water*
Section in the walk-in cooler indicated w/CLP sign-in, sign-out sheets	All samples that are under internal COC. CLP samples are also stored in here.

4.4.4.1. *See Section 4.7 for transfer of extractable aliquots to the extractions lab

4.4.4.2. Prior to putting bottles into any storage location, the electronic Bottle Summary Log must be completed. For Navy projects, unique bottle identifiers must also be entered in the bottle log.

4.4.4.3. At the J1 prompt, type "BOTTLES." This log details how many bottles were received, what type and size of bottles were received, the storage location of the bottles and the bottle numbers, where applicable. An example of this log follows.



4.5. Determination of tests

4.5.1. If Laucks provided sample bottles for the client, the bottle order, the client COC, file notes, letters, client instructions, or the client file are consulted as necessary to determine what tests are to be performed. The type of bottles received for water samples will help greatly in determining which tests to perform. If you can't determine the tests, give the paperwork to the Project Manager, who will contact the client.

4.5.2. A lab work request is initiated at this time. Based on a review of the above information, test codes are assigned to the appropriate fractions. These test codes may represent single-data point analyses ("regular" tests) or multiple-data point analyses ("special" tests), such as GC/MS volatiles. However, no work request packet can be prepared until after "transmittal," which is initiated by the Project Manager or designee.

4.5.3. Some soil samples will need to be shared between two or more departments. In order of priority, the following areas will receive samples in this order:

- If volatiles are requested, then the VOA departments will get the samples first (GC or GC/MS)
- The sample/samples will then be sent to the extractions lab
- The extractions lab will return the sample/samples to the inorganics lab or other areas

At the time of sample log-in the Sample Custodian will make appropriate comments for the department to return the samples to other departments for further testing. **Before** any samples are sent to other departments for testing, it is imperative that any requiring the analysis of volatile organics gets the sample first. Such samples should be given to the Volatiles Department before any other department.

4.6. Electronic Transmittal of Sample and Test Request Records

4.6.1. Specifics of the transmittal process are detailed in a separate SOP. A brief summary follows. For actual transmittal, that SOP should be referenced as it will detail greater specifics and will contain changes that may occur in the transmittal process. The following is only intended as a brief overview and may not reflect the most current practices.

4.6.2. All documentation (including, but not limited to, air bills, chain-of-custody documents, bottle order forms, notes, contracts, messages, letters, etc.) that supports the information entered into the work order and the sample fractions is clipped together by the Sample Custodian when sample log-in is complete. The work order number is written on, at a

minimum, the chain-of-custody document and may also be written on any other relevant documents.

4.6.3. The supporting documentation is given to the project manager, her designee, or to the head of the Project Management Group for "transmittal." Transmittal is the electronic approval of the work order and sample fractions as written and must be performed within 1 working day of sample log-in. Transmittal is the activity which electronically puts the samples and test requests into the laboratory's analytical schedule.

4.6.4. In performing the transmittal, it is the responsibility of the Project Manager, or designee, to double-check the work order and test fractions for the following:

- accuracy of project information (number, name, point of contact, etc.)
- accuracy of test requests
- and accuracy of the test codes employed to represent those test requests.

The Project Manager makes corrections to these items as necessary, usually in consultation with the Sample Custodian. When transmittal is complete, the hard-copy record generated in the transmittal process is stapled to the supporting documentation previously assembled by the Sample Custodian and the complete record is filed alphabetically (by client name) in the filing drawer designated. If a CLP-style package is being generated, packets are prepared for the CLP Document Control Custodian.

4.6.5. Specific test requests are made known to analysts through hard-copy work "backlogs". For a description of this process, see the SOP on Data Handling.

4.7. Generation of internal Chain-of-Custody (COC)

4.7.1. Samples which must be removed from the main building at 940 and taken to the extractions lab at 921 for preparation are tracked with an internal COC. This form is initiated by the person logging in the samples. The lab number, the client name, the number of samples, the sample matrixes and the enterer's initials and the date and time the form is started are recorded. See Appendix 4 for an example of an internal COC.

4.7.2. The samples are placed on shelf 8C in the walk-in with the COC. The person removing the samples from 940 signs and dates the form. The samples are logged into a log book at 921 before being placed in the cooler.

4.7.3. The COC is returned to 940 with the extracts when extractions are completed.

4.8. Sample breakage

4.8.1. All sample breakage, whether in shipping or while handling in the lab, must be reported immediately to the Project Manager.

4.8.2. If the sample was water, clean thoroughly with disposable towels. Be very careful with broken glass so as to avoid cuts.

4.8.3. If the sample was soil, as much of the sample as possible is transferred to a new, clean jar using a spatula. Be certain not to pick up any sample which has contacted the floor. Save the original label, if possible. Note on the log-in records that the sample was broken and transferred to a new container.

4.8.4. All dirty, disposable clean up materials, soil, broken glass, etc. are placed in a plastic garbage bag before being placed in the dumpster. Any non-disposable clean up materials are washed after use.

4.9. Special circumstances

4.9.1. Samples from some clients are logged into a monthly work order. Some jobs extending over more than one sampling event may be entered under one work order number. In that event, pay special attention to date of receipt (see ALERT, above).

4.9.2. Other special circumstances may arise. If there are any questions, check with the Project Manager first.

APPENDIX 1

Sample Receipt Verification Stamp

Sample Receipt acknowledged pending verification of sample count. You will be notified within one working day of any discrepancies found

Signed _____ Date _____ Time _____

APPENDIX 2

In-House Sample Receipt Log

Laucks Testing Laboratories, Inc.
IN-HOUSE SAMPLE RECEIPT LOG

DATE RECEIVED:_____ WORKORDER #:_____
CLIENT NAME:_____
RECEIVED BY:_____

Non-Conformance:

☐ Other

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

CORRECTIVE ACTION: (Check applicable item(s))

		Correction action taken by: Inititals	Date
☐	(1) Client informed verbally (Client Services).	_____	
☐	(2) Client informed by memo/letter/fax (Client Services).	_____	_____
☐	(3) Sample processed "as received" (Sample Entry).	_____	_____
☐	(4) Re-sampling requested of client (Client Services).	_____	_____
☐	(5) Samples placed "on hold" until further notice (Sample Entry/Client Services).	_____	_____

* When complete (within 24 hours of nonconformance) forward to QA. Original to be forwarded to initiator to be included in transmittal file.

Comments:

_____

_____

_____

_____

_____

APPENDIX 3

CLP Sample Receipt Log
Supplemental Sample Receipt Log

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

P:\PM\Holly\docs\fax\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

Work Order Number:_____
Assigned SDG Number:_____

Temperature		pH of Bottle Types								

Allowable temperature and pH ranges (neutral pH defined as a value between 5 and 9)

Temperature	Allowable temperature range is 4± 2 degrees Celsius
Acid Preserved pH	pH must be less than 2
Base Preserved pH	pH must be greater than 12

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.

Secure Storage Custody Log

ect: _____ 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 or Method 8260/Method 8240, GC/MS 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	
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 or Method 8260/Method 8240, GC/MS 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	

Laucks Testing Laboratories, Inc.

B. WASTE EVALUATION - ORGANICS ANALYSIS (SW-846, 3rd Edition) (continued)

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Aromatic Volatile Organics	2 - 40 ml containers for liquids 20 grams for solids	Glass, Teflon-lined Septum, 40 ml capacity Above or Glass, 2-4 oz. capacity	Cool, 4° C, if there is presence of residual chlorine then preserve with 0.5 g ascorbic acid and adjust pH to ≤2 with HCl, no headspace Cool, 4° C	14 days with preservation 14 days	Method 8020, GC/PID Direct Injection or Headspace, Method 5020 - Purge-and-Trap, Method 5030 Method 8260/8240, GC/MS Purge-and-Trap Method
Acrolein, Acrylonitrile Acetonitrile	2 - 40 ml containers for liquids 20 grams for solids	Glass, Teflon-lined Septum, 40 ml capacity Above or Glass, 2-4 oz. capacity	Cool, 4° C, adjust pH 4-5 with HCl, no headspace Cool 4° C, no headspace	14 days, with preservation 14 days	Method 8030, GC/FID Direct Injection or Headspace, Method 5020 - Purge-and-Trap Method 5030 - Groundwater using Method 5030 only. Method 8260/8240, GC/MS Purge-and-Trap Method
Phenols	Approximately 1 liter for liquid sample Approximately 50 grams for sludge or solid sample	Glass, Teflon-lined cap	Cool, 4° C, 35 mg $Na_2S_2O_3$ per ppm free chlorine per liter, adjust pH <2 with H_2SO_4 Cool 4° C	Extracted within 7 days and completely analyzed within 40 days Extracted within 14 days and completely analyzed within 40 days	Method 8040 GC/FID or GC/ECD or Method 8270 GC/MS
Organochlorine Pesticides and PCBs	Approximately 1 liter for liquid sample Approximately 50 grams for sludge or solid sample	Glass, Teflon-lined cap	Cool, 4° C, adjust pH to 6-8 with H_2SO_4 or NaOH Cool, 4° C	Extracted within 7 days and completely analyzed within 40 days Extracted within 14 days and completely analyzed within 40 days	Method 8080 GC/ECD

B. WASTE EVALUATION - ORGANICS ANALYSIS (SW-846, 3rd Edition) (continued)

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Polynuclear Aromatic Hydrocarbons (PAHs)	Approximately 1 liter for liquid sample Approximately 50 grams for sludge or solid sample	Glass, Teflon-lined cap	Cool, 4° C	Extracted within 7 days and completely analyzed within 40 days Extracted within 14 days and completely analyzed within 40 days	Method 8310 HPLC or Method 8270 GC/MS
Chlorinated Hydrocarbons	Approximately 1 liter for liquid sample Approximately 50 grams for sludge or solid sample	Glass, Teflon-lined cap	Cool, 4° C	Extracted within 7 days and completely analyzed within 40 days Extracted within 14 days and completely analyzed within 40 days	Method 8270 GC/MS
Organo-phosphorus Pesticides	Approximately 1 liter for liquid sample Approximately 50 grams for sludge or solid sample	Glass, Teflon-lined cap	Cool, 4° C	Extracted within 7 days and completely analyzed within 40 days 14 days	Method 8140 GC/NPD or GC/NPD/ECD
Chlorinated Herbicides (i.e., 2,4-D and 2,4,5-TP)	Approximately 1 liter for liquid sample Approximately 50 grams for sludge or solid sample	Glass, Teflon-lined cap	Cool, 4° C	Extracted within 7 days and completely analyzed within 40 days Extracted within 14 days and completely analyzed within 40 days	Method 8150 Extraction and Esterification/GC-ECD

Laucks Testing Laboratories, Inc.

B. WASTE EVALUATION - ORGANICS ANALYSIS (SW-846, 3rd Edition) (continued)

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Volatile Organics (VOAs)	2- 40 ml containers for liquid sample 20 grams for solids	Glass, Teflon-lined Septum 40 ml capacity As above or glass, 2-4 oz.	Cool, 4° C, acid preserved with HCl to pH < 2, no headspace Cool, 4° C, no headspace or if solid packed to minimize headspace	14 days 14 days	Method 8260 Purge-and-Trap GC/MS
Semi-Volatile Organics	Approximately 1 liter for liquid sample Approximately 50 grams for sludge or solid sample	Glass, Teflon-lined cap	Cool, 4° C,	Extracted within 7 days and completely analyzed within 40 days Extracted within 14 days and completely analyzed within 40 days	Method 8270 GC/MS

C. WASTE EVALUATION - GENERAL

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Toxicity Characteristic Leaching Procedure	Approximately 1 liter for liquid sample Approximately 200 grams for solid sample	Glass, Teflon-lined cap	VOA, Metals, Semivolatiles, Pesticides/Herbi[illegible] 14 days until extracti[illegible] Follow analytical protocol for aqueous holding time or holding time from leachate preparation.	Not specified	According to requested analysis
Water Reactivity	Approximately 100 ml for liquid sample Approximately 50 grams for solid sample	----	None	14 day	----

C. WASTE EVALUATION - GENERAL (continued)

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Ignitability	Approximately 200 ml or 50 grams	None specified	None	14 days	Method 1010 Pensky-Martens Closed-Cup Method Method 1020 Setaflash Closed Cup Method
Corrosivity	100 - 500 ml	Plastic	None	None	Method 1110 Corrosivity Toward Steel
California Assessment Manual CAM/STLC	Approximately 200 ml for liquid sample Approximately 10 grams for solid sample	Plastic or Glass	No preservation for solid sample. Add HNO_3 to pH <2 for liquid sample	28 days for Mercury 6 months for others	See Methods for Metals Analysis

D. METALS ANALYSIS (EPA Methods for Chemical Analysis of Water and Wastes, March 1983, or APHA Standard Methods, 15 Edition. and EPA SW-846, 3rd Edition)

PARAMETER	MINIMUM VOLUME REQUIRED **	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Mercury, Total/Dissolved	100-200 ml for liquid sample	Plastic or Glass	HNO_3 to pH <2 for total	28 days	EPA 245.1 for water or EPA 7470A
Mercury, Total	Approximately 5 grams for solid sample		Filter on site, HNO_3 to pH <2 for dissolved		EPA 7471A for sediment Cold Vapor Method
Metals, Total Metals, Dissolved	300 ml for liquid samples Approximately 10 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 for total Filter on site, HNO_3 to pH <2 for dissolved	6 months	Flame AA-- See Individual Metal Methods Emission AA-- See Individual Metal Methods Graphite Furnace AA- See individual Metal Methods or ICP-- 200.7 or 6010

** For individual metals the aggregate minimum volume is determined by the number of discrete analytical methods not the sum of all the individual analytes.

D. METALS ANALYSIS (EPA Methods for Chemical Analysis of Water and Wastes, March 1983, or APHA Standard Methods, 15 Edition. and EPA SW-846, 3rd Edition) (continued)

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Hexavalent Chromium -Cr^6	200 ml for liquid sample Approximately 50 grams for solid samples	Plastic or Glass	Cool, 4 C	24 hours Extracted within 7 days, analyzed within 24 hrs. of extraction.	EPA 218.4 or EPA 7196A EPA 218.5 or EPA 7197 Extraction/AA Method EPA 7196 Colorimetric Method
Aluminum (Al)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 202.1, Flame EPA 202.2 or EPA 7020, Furnace
Antimony (Sb)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 204.1 or EPA 7040, Flame EPA 204.2 or EPA 7041, Furnace
Arsenic (As)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 206.3 or EPA 7061, Hydride AA EPA 206.2 or EPA 7060, Furnace AA
Barium (Ba)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 208.1 or EPA 7090, Flame AA EPA 208.2, Furnace AA
Beryllium (Be)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 210.1 or EPA 7090, Flame AA EPA 210.2A or EPA 7091, Furnace AA
Boron (B)	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic only	Cool, 4° C	6 months	Curcumin Colorimetric EPA 212.3

** Each metal can also be analyzed by EPA 200.7, EPA 6010A, or EPA 6020.

D. METALS ANALYSIS (EPA Methods for Chemical Analysis of Water and Wastes, March 1983, or APHA Standard Methods, 15 Edition. and EPA SW-846, 3rd Edition) (continued)

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Cadmium (Cd)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 213.1 or EPA 7130, Flame AA EPA 213.2 for EPA 7131, Furnace AA
Calcium (Ca)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 215.1 or EPA 7140, Flame AA
Chromium (Cr)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 218.1 or EPA 7190, Flame AA EPA 218.2 or EPA 7191, Furnace AA EPA 218.3 or EPA 7198, Chelation Extraction
Cobalt (Co)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 219.1 or EPA 7200, Flame AA EPA 219.2 or EPA 7201, Furnace AA
Copper (Cu)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 220.1 of EPA 7210, Flame AA EPA 220.2, Furnace AA
Gold (Au)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 231.1, Flame AA EPA 231.2, Furnace AA
Iron (Fe)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 236.1 or EPA 7380, Flame AA EPA 236.2, Furnace AA

** Each metal can also be analyzed by EPA 200.7, EPA 6010A, or EPA 6020.

D. METALS ANALYSIS (EPA Methods for Chemical Analysis of Water and Wastes, March 1983, or APHA Standard Methods, 15 Edition. and EPA SW-846, 3rd Edition) (continued)

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Lead (Pb)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 239.1 or EPA 7420, Flame AA EPA 239.2 or EPA 7421, Furnace AA
Magnesium (Mg)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 242.1 or EPA 7460, Flame AA
Manganese (Mn)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 243.1 or EPA 7480, Flame AA EPA 243.2 or EPA 7481, Furnace AA
Molybdenum (Mo)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 246.1 or EPA 7520, Flame AA EPA 246.2, Furnace AA
Nickel (Ni)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 249.1 or EPA 7610, Flame AA EPA 249.2, Furnace AA
Potassium (K)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 258.1, Flame AA
Selenium (Se)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 270.2 or EPA 7740, Furnace AA EPA 270.3 or EPA 7741, Hydride AA

** Each metal can also be analyzed by EPA 200.7, EPA 6010A, or EPA 6020.

D. METALS ANALYSIS (EPA Methods for Chemical Analysis of Water and Wastes, March 1983, or APHA Standard Methods, 15 Edition. and EPA SW-846, 3rd Edition) (continued)

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Silver (Ag)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 272.1 or EPA 7760, Flame AA EPA 272.2, Furnace AA
Sodium (Na)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 273.1 or EPA 7770, Flame AA
Thallium (Tl)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 279.1 or EPA 7840, Flame AA EPA 279.2 of EPA 7841, Furnace AA
Tin (Sn)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 282.1 or EPA 7870, Flame AA EPA 282.2, Furnace AA
Titanium (Ti)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 283.1, Flame AA EPA 283.2, Furnace AA
Vanadium (V)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 286.1 or EPA 7910, Flame AA EPA 286.2 or EPA 7911, Furnace AA
Zinc (Zn)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 289.1 or EPA 7950, Flame AA EPA 289.2, Furnace AA

** Each metal can also be analyzed by EPA 200.7, EPA 6010A, or EPA 6020.

E. GENERAL: MINERAL ANALYSIS/VOLATILES - DRINKING WATER or TITLE 22 CAL/DOHS

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Complete General Mineral Analysis	1 - 2 liters	Plastic or Glass	Cool, 4° C, additional preservation depends on the analyte list	----	----
pH	50 ml	Plastic or Glass	None	Immediate	EPA Method 150.1 pH Meter
Alkalinity	50 - 100 ml	Plastic or Glass	Cool, 4° C	14 days	EPA Method 310.1 Titrimetric Method
Calcium**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	Flame AA EPA 215.1
Chloride	50 - 100 ml	Plastic or Glass	Cool, 4° C	28 days	EPA Method 325.3 Titrimetric Method
Copper**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	Flame AA EPA 220.1
MBAS	500 ml	Plastic or Glass	Cool, 4° C	48 hours	EPA Method 425.1 Colorimetric
Iron**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	Flame AA EPA 236.1
Magnesium**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	Flame AA EPA 242.1
Manganese**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	Flame AA EPA 243.1
Sodium**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	Flame AA EPA 273.1
Sulfate	50 - 100 ml	Plastic or Glass	Cool, 4° C	28 days	EPA 375.4 Turbidimetric
Electrical Conductivity	50 - 100 ml	Plastic or Glass	Cool, 4° C	24 hrs. or filter	EPA Method 120.1 EC Water
Total Dissolved Solids	100 ml	Plastic or Glass	Cool, 4° C	7 days	EPA Method 160.1 Gravimetric
Total Hardness	50 - 100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	EPA Method 130.2 Titrimetric Standard Method 314-A Calculation
Zinc**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	Flame AA EPA 289.1
Volatile Organics (VOAs)	2 - 40 ml containers	Glass, Cap teflon-lined, 40 ml.	No headspace, HCl to pH<2, if residual chlorine then preserve with Na_2SO_4 or Ascorbic Acid Wash. State - No headspace and HCl to pH <2 only	14 days	EPA Method 524.2

** Each metal can also be analyzed by EPA 200.7 or EPA 6010A.

F. INORGRANIC ANALYSIS: DRINKING WATER or TITLE 22 - CAL/DOHS

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Arsenic**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	EPA 206.3, Furnace AA
Barium**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	EPA 208.1, Flame AA
Cadmium**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	EPA 213.1 , Flame AA or EPA 218.2, Furnace AA
Chromium**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	EPA 218.1, Flame AA EPA 218.2, Furnace AA
Lead**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	EPA 239.2, Furnace AA
Mercury	100 ml	Plastic or Glass	HNO_3 to pH <2	28 days	EPA 245.1 ,Cold Vapor
Selenium**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	EPA 270.2 ,Furnace AA
Silver**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	EPA 272.1 ,Flame AA
Nitrate-Nitrogen	50 ml	Plastic or Glass	Cool, 4° C add H_2SO_4 to pH <2	14 days	EPA 352.1, Brucine Sulfate
			Cool, 4° C	48 hours	EPA 353.3, Cadmium Reduction
Fluoride	300 ml	Plastic or Glass	None	28 days	EPA 340.2, Ion Selective Electrode

** Each metal can also be analyzed by EPA 200.7 or EPA 200.8.

G. GENERAL PHYSICAL ANALYSIS: DRINKING WATER or TITLE 22 - CAL/DOHS

PARAMETER	VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Color	50 ml	Plastic or Glass	Cool, 4° C	48 hours	EPA Method 110.2
Odor	200 ml	Plastic or Glass	Cool, 4° C	48 hours	EPA Method 180.1, Threshold Odor
Turbidity	100 ml	Plastic or Glass	Cool, 4° C	48 hours	EPA Method 180.1, Nephelometric

H. GENERAL WET CHEMISTRY AND MISCELLANEOUS ANALYSIS

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Acidity	100 ml	Plastic or Glass	Cool, 4° C	14 days	EPA 305.1, Titrimetric
Alkalinity	50-100 ml	Plastic or Glass	Cool, 4° C	14 days	EPA 310.1 ,Titrimetric
Ammonia Nitrogen	100 ml	Plastic or Glass	Cool, 4° C H_2SO_4 to pH <2	28 days	EPA 350.1, Colorimetric
BOD	1 liter	Plastic or Glass	Cool, 4° C	48 hours	EPA 405.1
Boron	100 ml	Plastic	None	28 days	EPA 212.3 ,Curcumin
Chloride	100 ml	Plastic or Glass	None	28 days	EPA Method 325.3, Mercuric Nitrate or EPA 300.0, Ion Chromatography
COD	20 ml	Plastic or Glass	Cool, 4° C H_2SO_4 to pH <2	28 days	EPA Methods 410.4, Colorimetric
Coliform Fecal Coliform	100 ml	Sterilized Plastic Bottles	Cool, 4° C $Na_2S_2O_3$ preserved for presence of free chlorine	6 hours or 30 hours depending on the test requested	Standard Method 909A or 909C
Color	50 ml	Plastic or Glass	Cool, 4° C	48 hours	EPA 110.2 or 110.3
Cyanide	500 ml	Plastic or Glass	Cool, 4° C NaOH to pH >12	14 days	EPA 335.3, Colorimetric
Electrical Conductivity	50 - 100 ml	Plastic or Glass	Cool, 4° C	28 days	EPA 120.1, EC Meter
Fluorides	300 ml	Plastic or Glass	None	28 days	EPA 340.2, Ion Specific Electrode or EPA 300.0, Ion Chromatography
MBAS	500 ml	Plastic or Glass	Cool, 4° C	48 hours	EPA 425.1, Colorimetric

H. GENERAL WET CHEMISTRY AND MISCELLANEOUS ANALYSIS (continued)

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Nitrate Nitrogen	100 ml	Plastic or Glass	Cool, 4° C	14 days	EPA 300.0, Ion Chromatography
Nitrate Nitrite Nitrogen	100 ml	Plastic or Glass	Cool, 4° C Add H_2SO_4 to pH <2	14 days	EPA 353.3, Cadmium Reduction
Nitrite Nitrogen	50 ml	Plastic or Glass	Cool, 4° C	48 hours	EPA 354.1, SPectrophotometric
Odor	200 ml	Plastic or Glass	Cool, 4° C	24 hours	EPA 140.1
Oil and Grease	1 liter	Glass	Cool, 4° C Add H_2SO_4 to pH <2	28 days	EPA 413.1, Gravimetric
Orthophosphate	50 ml	Plastic or Glass	Cool, 4° C	48 hours	EPA 365.2, Ascorbic Acid
pH	50 ml	Plastic or Glass	None	Immediate	EPA Method 150.1, pH Meter
Phenolics	500 ml	Glass	Cool, 4° C Add H_2SO_4 to pH <2	28 days	EPA 420.1, 4-AAP
Radioactivity	1-18 liters	Plastic or Glass Glass only for Tritium	HCl or HNO_3 * to pH <2 *Some exceptions	----	Standard Method 701
Silica	50 ml	Plastic	Cool, 4° C	28 days	EPA 370.1, Colorimetric Flame AA Method
Sulfates	50 ml - 100 ml	Plastic or Glass	Cool, 4° C	28 days	EPA 375.4, Turbidimetric or EPA 300.0, Ion Chromatography
Sulfides	500 ml	Plastic or Glass	NaOH to pH >9 2 ml Zinc Acetate Cool, 4° C	7 days	EPA 376.1 ,Titrimetric
Sulfites	50 ml	Plastic or Glass	None	Immediate	EPA 377.1 ,Titrimetric
TOC	25 ml	Plastic or Glass	Cool, 4° C H_2SO_4 to pH <2	28 days	EPA 415.2, TOC Analyzer
Total Dissolved Solids	100 ml	Plastic or Glass	Cool, 4° C	7 days	EPA 160.1, Gravimetric

H. GENERAL WET CHEMISTRY AND MISCELLANEOUS ANALYSIS (continued)

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Total Hardness	50-100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	EPA 130.2, Titrimetric Standard Method 314-A Calculation
Total Kjeldahl Nitrogen	1 liter	Plastic or Glass	Cool, 4° C Add H_2SO_4 to pH <2	28 days	EPA 351.4, Ion Specific Electrode
Total Organic Nitrogen TON-TKN-NH_3-N	1 liter	Plastic or Glass	Cool, 4° C Add H_2SO_4 to pH <2	28 days	EPA 351.4 and EPA 350.3
Total Petroleum Hydrocarbons Scan, by GC	400 ml to 1 liter Approximately 50 grams for a solid sample	Glass	Cool, 4^0 C	14 days, but this may vary between states so regulations must be consulted	WTPH-HCID by GC/FID or Modified Method 8015, GC
Total Petroleum Hydrocarbons as Gas, by GC	40 ml Approximately 20 grams for a solid sample	Glass	Cool, 4^0 C, Methanol preservation may be a requirement of some states so state regulations must be consulted Cool, 4^0 C	14 days	WTPH-G by purge and trap GC/FID or Modified Method 8015, GC/FID
Total Petroleum Hydrocarbons as Diesel, by GC	1 liter Approximately 50 grams for a solid sample	Glass	Cool, 4^0 C	14 days	WTPH-D by GC/FID or Modified Method 8015, GC/FID
Total Phosphate	50 ml	Plastic or Glass	Cool, 4° C Add H_2SO_4 to pH <2	28 days	EPA 365.4, Colorimetric
Total Residue	100 ml	Plastic or Glass	Cool, 4° C	7 days	EPA 160.3, Gravimetric
Total Settleable Solids	1 liter	Plastic or Glass	Cool, 4° C	48 hours	EPA 160.5, Imhoff Cone
Total Suspended Solids	100 ml	Plastic or Glass	Cool, 4° C	7 days	EPA 160.2, Gravimetric
Total Volatile Solids	100 ml	Plastic or Glass	Cool, 4° C Add H_2SO_4 to pH <2	7 days	EPA 160.4, Gravimetric
TOX	500 ml	Amber Glass, Teflon Septum	Cool, 4° C Add H_2SO_4 to pH <2	7 days	EPA 450.1 or EPA 9020, TOX Analyzer
Turbidity	100 ml	Plastic or Glass	Cool, 4° C	48 hours	EPA 180.1

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-4103

Title: Reviewing a Sample Delivery Group (SDG) and Updating Projqc in the LIMS

Revision History:

Number	Date
0	4/09/96

Written by: Diana Spence Date: 4/9/96
Diana Spence

Reviewed by: Harry Romberg Date: 4-9-96
Harry Romberg, QC Officer

Approved by: Karen J. Kotz Date: 4/9/96
Karen Kotz, Laboratory Director

UNCONTROLLED

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1. Introduction and Scope

1.1. Method Description

1.1.1 The project manager (PM) or their designee reviews the LIMS computer entry versus the chain-of-custody (COC) for accuracy. This should be done as soon as practical and within twenty-four hours (24) of sample receipt.

1.1.2 This method is restricted to use by the person (usually a project manager) who performed the work order transmittal.

1.2. Definition of Terms

1.2.1. SDG - Sample Delivery Group

Projqc - Project QC section of the LIMS. This is where all of the work orders for a specific project are listed with additional information about the QC requirements, TATs and deliverables.

2. Equipment List

2.1. Equipment

2.1.1. The person performing this procedure must have access to a computer which is connected to the LIMS.

3. Operation procedures

3.1. Reviewing the SDG Entry in LIMS

3.1.1 Once a work order has been transmitted, go to the SDG section of the LIMS. This is done by entering [SDG, space, SDG name] at the J(1): prompt. The SDG name is found in the fractions sections of the work order in the SDG field labeled 'SDG#'. **Example** - J(1): SDG HCST4. See Appendix I for an example.

3.1.2 In the SDG verify that all the samples have been entered and that the correct 'fraction' of the SDG (I,G, V, P, etc.). The letters refer to the type of analysis, i.e., Inorganics, GC, Volatiles, and PAHs. These are a few of the types of

fractions which might be contained in a SDG. It is not important to know the abbreviations. However, it should be verified that all analyses for each sample can be found in the SDG by looking at the various fractions for that SDG. A fraction may contain more than one analysis. For instance an 'I' fraction (inorganics) might have TOC, Metals and TSS.

3.1.3 When the SDG name is entered at the J(1): prompt, the 'first' page of the first fraction of a SDG will be displayed. The first fraction is determined alphabetically based upon the letter associated with an analysis. For instance, if a work order has analyses for GC analysis (G), metals (I) and PAHs (P), the fractions of a SDG would be listed in LIMS with GC analyses first, metals second, and PAHs last. The information contained on the first page is:

- Laucks sample ID
- QC Designations
- Client ID
- Matrix

3.1.4. Verify that all the samples for that fraction (analysis) have been entered. this is facilitated by entering [F6] which displays the second , third and fourth pages of a fraction. [F6] is a toggle key which pages through the four screens of each SDG entry. The far left hand column of the computer screen will always display the lists of the SDG names with the fractions appendix letter. **Example** - GSI01G (GC fraction), GSI01I (inorganics fractions). See pages 1,2 and 3 of Appendix 1 for examples of each page of a SDG.

3.1.5 The second page of a SDG contains the Laucks samples ID, the VTSR (Verified Time of Sample Receipt) and date collected information.

3.1.6 The third page of a SDG contains the Laucks sample ID and the specific analysis requested. It is important to review this page carefully. If there are many samples in that fraction, they may not all be visible at once. Move down the screen to view all samples by repeatedly pushing the down arrow key, [↓].

3.1.7 Once this fraction has been checked, the other fractions can be checked by using the [F3] (moves the cursor up) and [F4] (moves the cursor down) function keys to place the cursor on the next fraction of the SDG. It is displayed in the column on the left of the screen.. It is possible to view the same page of different fractions by just moving the cursor to the next fraction. For example, if you are in page three of a fraction, when you move to the next fraction, page three of this fraction will

be displayed. **[F6] controls the page selection of the SDG, [F3] and [F4] respectively in the far left column.**

3.1.8 If corrections are necessary, return the COC and log-in documents to the Sample Control department. If the person performing the review makes the corrections, the changes must be saved by entering [F8].

3.1.9 To exit the SDG, enter [F1] until the J(1): prompt is displayed.

3.2 Entering a Work Order into 'Projqc' of LIMS

3.2.1 At the J(1): prompt, enter [projqc, space, projqc name]. Example - J(1): projqc OHM_Hawaii. If you are not sure of the projqc name, a name may be entered which is similar. This will bring up the projqc in the vicinity of the name which is desired. The cursor is moved up, [F3], or down, [F4}, to the desired name. The projqc names will be displayed in the far left column of the screen. See page 1 of Appendix II.

3.2.2 Once the correct project name is displayed, move the cursor down to the next empty line in the work order column. enter the work order number. The SDG will appear in the SDG list column. Move the cursor to the matrix columns (W = Water, S = Soil, O = Oil). Under the appropriate column, enter the number of samples for each matrix. If the SDG is to remain open, no other information should be added. Enter [F8] to save the updated SDG.

3.2.3 It the SDG is to be closed, the due date for the data to be submitted to the reporting department and the due date for the hardcopy report to the client must be entered into the appropriate columns. These dates are displayed in Projqc as 'Office' and 'Client' respectively. See page 1 of Appendix II. Enter [F8] to save these changes.

3.2.4 SDG closure is determined by several factors:

- The number of samples in the SDG, per the EPA definition, should not contain more than twenty samples of the same matrix received over a period of not more than 14 calendar days.

- If it is known that more samples will be arriving for the same project in less than fourteen days, and the SDG is not full, it may be desirable to maintain the SDG in 'open' status until arrival of the next samples.

- If the client desires a fast TAT for the hardcopy package, the SDG should be closed regardless of the number of samples in it.

These are judgment decisions with the exception of the EPA definition of an SDG), which must be made by the project manager.

3.2.5 Once the SDG has been closed, send e-mail notification of the closure to all departments affected. This would be any department involved with the analyses for the work order, Sample Control and the reporting department.

3.2.6 Finally, the paperwork is submitted to the reporting department detailing the nature and status (open or closed) of an SDG. The forms submitted would be the following, arranged in the order listed below:

- Pre-package checklist
- Chain-of-Custody forms (original or top, white copy)
- * Sample Receipt Log (1) CLP (original)
- * Supplemental Sample Receipt Log (original)

* A copy of each of these forms must be made and attached to the copy of the COC for the transmittal.

See Appendix III for examples of these forms.

3.3 Creating a New Projqc

3.3.1 When a new project commences, it may be necessary to create a new entry in Projqc. The factors which would determine if this is necessary may include the following:

- The complexity of the project
- The duration of the project
- How many laboratory departments are affected by the project

Projqc entries are applicable to both CLP and non-CLP project. The information contained in this section of the LIMS is accessible by all laboratory staff who have rights to the LIMS and is a valuable form of communication for project requirements. However, it does not take the place of kick-off meetings for projects or e-mail regarding specific samples problems.

3.3.2 To create a new projqc entry, enter [projqc] at the J(1): prompt. This will display the first projqc entry in the LIMS. The cursor will be in the field called 'Proj Name:'. Enter the name of the new projqc entry. this name may consist of up to ten characters, alpha and numeric, and one underscore in the place of a hyphen, if desired. Once entered, push [Enter]. The cursor will move to the next field, 'Client Code'. See page 1 of Appendix II.

3.3.3 In the 'Client Code' field, enter the correct client code for this work order. This can be found in the work order. Push [Enter]. The cursor will next appear in the 'Desc:' field.

3.3.4 In this field enter a brief description (one cryptic sentence) of the project. Push [Enter]. the cursor will next appear in the bottom line of the projqc header. Enter the correct information for these questions (QAPP?, pH/Temp?). This would usually be 'yes' if the project is CLP or USACE (US Army Corps of Engineers).

3.3.5 Due to software complications, in the field 'Sort by (W/S):' it is best to enter [N].

3.3.6 Enter [F8] to save this new projqc. Item 3.3.1 through 3.3.5 would be considered the header information for a projqc. This is page 1 of the projqc. It is here that the work orders for that project will be entered when the samples arrive at the laboratory.

3.3.7 Next go to page 2 of the projqc. This is accomplished by pushing [F6]. The header information will already be there. The information which must be entered here is:

- Results TAT (Fax)
- Report TAT (hardcopy)
- Deliverables - CLP, LTL (Laucks Testing Laboratory or non-CLP)
- MPR - Monthly Progress Report (required for HAZWRAP projects)
- EDD deliverables - CLP or LTL (any non-CLP, custom EDD)
- Deliverables Comments - Any special project comments
- Penalties - enter any applicable penalties here

Enter [F8] to save this information.

3.3.8 Next go to page three of the projqc. This is accomplished by entering [Ctrl,F6]. of a project can be written as an overview. See page three of Appendix II. The information contained here is:

- Client name
- Project Name
- Overview
- Schedule
- Analytes
- Protocol
- QC
- Turnaround
- Penalties
- CRDLs
- Holding Times
- Deliverables
- Additional Comments

After entering this information, enter [F8] to save it. It may be advantageous to enter [F8] periodically as the information is entered to prevent loss in the event of a power surge.

Appendix I

Example of SDG Entry in LIMS

GSI01
KEYS
GLS08S
GLS08T
GLS08V
GLS09E
GLS09I
GLS09O
GLS09P
GLS09S
GLS09T
GLS09V
GSI01G
GSI01I
GSI01P
GSI01V
GSI02G
GSI02I
GSI02P
GSI02V
GSI03G

SDG Database

SDG Group : GSI01 Date Due: 11/10/95 Created: 10/12/9
Fraction : G SPVMITO Updated: 10/23/9
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		B1018GSVSLV
9510423	02		B952/SB-952-1/SS9,16-18/LK	S	89		B1018GSVSLV
9510423	03		B952/SB-952-2/SS4,6-8/LK	S	83		B1018GSVSLV
9510423	04		B952/SB-952-2/SS5,8-10/LK	S	84		B1018GSVSLV
9510434	01		B952/SB-952-10/SS8,14-16LK	S	84		B1018GSVSLV
9510434	02		B952/SB-952-3/SS3,4-6/LK	S	84		B1018GSVSLV
9510434	03	MS	B 52/SB-952-3/SS6,10-12/LK	S	89		B1018GSVSLV
9510434	05	BMS	CH-5092500-S-SPOON-ERB-1	W			B1017GSVWLC
9510434	06		CH-POT-FB-1	W			B1017GSVWLC
9510434	07		CH-ASTM-5092500-FB-1	W			B1017GSVWLC
9510459	01		B952/SB-952-4/SS4,6-8/LK	S	84		B1018GSVSLV
9510459	02		B952/SB-952-4/SS7,12-14/LK	S	89		B1018GSVSLV
9510459	03		B952/SB-952-5/SS4,6-8/LK	S	87		B1018GSVSLV
9510459	04		B952/SB-952-5/SS6,10-12/LK	S	87		B1018GSVSLV
9510459	08		B952/SB-952-6/SS4,6-8/LK	S	84		B1018GSVSLV
9510459	09		B952/SB-952-6/SS6,10-12/LK	S	90		B1018GSVSLV
9510497	01		B952/SB-952-7/SS3,4-6/LK	S	89		B1018GSVSLV
9510497	02		B952/SB-952-7/SS8/4-16/LK	S	91		B1018GSVSLV
9510540	01		B952/SB-952-8/SS3,4.0-6.0	S	90		B1018GS
9510540	02		B952/SB-952-8/SS6,10.0-12	S	90		B1018GSVSLV

SDG Database Report
Page 2

Laucks Testing Labs

Report Date: 01/25/96

SDG Group : GSI01 Date Due: 11/10/95
Fraction : G
Project : AFCEE/Chanute 952 Client: GSI (TPH 8015) JP4
SAS Number: _____ Case Number: _____

Work Ord	Samp Num	VTSR	Date Collected
9510423-01		10/11/95	10/10/95
9510423-02		10/11/95	10/10/95
9510423-03		10/11/95	10/10/95
9510423-04		10/11/95	10/10/95
9510434-01		10/12/95	10/11/95
9510434-02		10/12/95	10/11/95
9510434-03		10/12/95	10/11/95
9510434-05		10/12/95	10/10/95
9510434-06		10/12/95	10/10/95
9510434-07		10/12/95	10/10/95
9510459-01		10/13/95	10/11/95
9510459-02		10/13/95	10/11/95
9510459-03		10/13/95	10/11/95
9510459-04		10/13/95	10/11/95
9510459-07		10/13/95	10/12/95
9510459-08		10/13/95	10/12/95
9510459-09		10/13/95	10/12/95
9510497-01		10/14/95	10/12/95
9510497-02		10/14/95	10/12/95
9510540-01		10/17/95	10/15/95
9510540-02		10/17/95	10/15/95

SDG Database Report
Page 3

Laucks Testing Labs

Report Date: 01/25/96

SDG Group : GSI01 Date Due: 11/10/95
Fraction : G
Project : AFCEE/Chanute 952 Client: GSI (TPH 8015) JP4
SAS Number: _____ Case Number: _____

Work Ord	Samp Num	Fractions JP4										
9510423-01		X										
9510423-02		X										
9510423-03		X										
9510423-04		X										
9510434-01		X										
9510434-02		X										
9510434-03		X										
9510434-05		X										
9510434-06		X										
9510434-07		X										
9510459-01		X										
9510459-02		X										
9510459-03		X										
9510459-04		X										
9510459-07		X										
9510459-08		X										
9510459-09		X										
9510497-01		X										
9510497-02		X										
9510540-01		X										
9510540-02		X										

Comments:

G
KEYS
080
8P
GLS08S
GLS08T
GLS08V
GLS09E
GLS09I
GLS09O
GLS09P
GLS09S
GLS09T
GLS09V
GSI01G
GSI01I
GSI01P
GSI01V
GSI02G
GSI02I
GSI02P

SDG Database

SDG Group : GSI01 Date Due: 11/10/95 Created: 10/12/95
Fraction : I SPVMITO Updated: 10/17/95
Project : AFCEE/Chanute 952 Client: GSI (Inorganic)
PROJQC ID : GS_CHANUTE Max. Samps: 21

Comments:
Use Control F6 for more PROJQC details.

F7=Del,/n,Name 8=Write,/n,In_use 9=Print,Db_enter?,Keys? 10=More,Help,Key

Appendix II

Example of Projqc in LIMS

Project QC Requirements Database

OHM
KEYS
C_MANANN
_MANANNS
C_MANANNW
KIC_ARCO
LOW_AFB_IN
LOW_AFB_OR
MCCHORDAFB
MCCHORDLTM
METRO_TBT
OHM-HAWAII
OHM-SITEA
OHM-SITEF
PASCO_AIR
PASCO_P
PASCO_S
PASCO_W
Q_CITY_F
SKAGIT
SODASPRING

Proj Name: OHM-HAWAII Client Code: OHM_HAWAII Created: 12/19/9
Desc: UST sites Updated: 01/22/9
QAPP? Y pH/Temp? Y Sort by (W/S): N By : DIANA

Ord#	Last Date	SDG#	Office	Client	Matrix W	Matrix S	Matrix O
9512516	12/19/95	HI033	01/31/96	02/02/96	2	3	
9601349	01/22/96	HI034	02/20/96	02/22/96	1	7	
9601351	01/10/96	HI035			3		
9601587	01/22/96	HI034	02/20/96	02/22/96	2	6	

F7=Del,,Name 8=Write,,In_use 9=Print,Help menu,PrtSc 10=More,Help,Key

OHM
KEYS
HC_MANANN
HC_MANANNS
HC_MANANNW
KIC_ARCO
LOW_AFB_IN
LOW_AFB_OR
MCCHORDAFB
MCCHORDLTM
METRO_TBT
OHM-HAWAII
OHM-SITEA
OHM-SITEF
PASCO_AIR
PASCO_P
PASCO_S
PASCO_W
Q_CITY_F
SKAGIT
SODASPRING

Project QC Requirements Database

Proj Name: OHM-HAWAII Client Code: OHM_HAWAII Created: 12/19/9
Desc: UST sites Updated: 01/22/9
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# GT127 open ☐ closed ☑

Work Order #(s)	Fax Due Date(s)
960312	1/16/96

Due to Office: 2/7/96

Due to client: 2/9/96

E.D.D.: yes ☐ Custom or CLP *(circle one)* no ☑

If yes, can the package ship without the disk? _____

Full Deliverables? yes ☑ no ☐

If no, what forms/raw data are required? _____

No. of Copies: 1

Special package requests:

Assigned Document controller: _____

THE 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): Daryl Brown (PRINTED NAME) Daryl Brown

WORK ORDER ID: 8 1-2153 PAGE 1 OF 1

DATE 1-8-96

SUBMITTED AT:

Laucks Testing Laboratories, Inc.

4101 South Hanford St., Seattle, WA 98108 (206) 767-5060 FAX 767-5063
1806 Terminal Way, Kalama, WA 98625 (360) 673-1605 FAX 673-2365

TESTS TO PERFORM: M8330

LAB SAMPLE	SAMPLE ID / LOCATION	DATE	TIME	M8330	NO. OF CONTAINERS	OBSERVATIONS, COMMENTS, SPECIAL INSTRUCTIONS
1)	INF-CL-209-010896	1-8-96	0900	2L	2L	Fax Results to 360-779-167

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

SUBMITTED VIA
[ ] UPS [ ] FED EX [ ] BUS
[ ] HAND [ ] _____

TEMPERATURE _____
[ ] AMBIENT [ ] REPRESENTATIVE

RELINQUISHED BY (SIGN AND PRINT)	DATE / TIME	RECEIVED BY (SIGN AND PRINT)	DATE / TIME
Daryl Brown	1-8-96 1600	Pony Express	
		PAM R. JOHNSON, LAUCKS TESTING LABS	1/9/96 0726

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

P:\PM\Holly\docs\labcomm.doc 12/2/94

Initial once samples are checked in _____

DATE RECEIVED: 1/9/96
TIME RECEIVED: 0726
CLIENT NAME: GSI
SDG # GTI27
COC # N/A

SAMPLE LOG-IN DATE: 1/9/96
WORKORDER #: 9601313
CLIENT PROJECT: Danger Site A
AIRBILL ATTACHED?:(#) 2657 6393
RECEIVED BY: B

Non-Conformance: (Check applicable item(s)) Client IDs affected:

- ☐ (1) Not enough sample sent for proper analysis. #s affected: _____
- ☐ (2) Sample Bottle received broken and/or cap not intact. _____
- ☑ (3) Custody seal: Absent ____ Present/Intact ✓ Present/Broken _____
- ☐ (4) Any temperature out of compliance: _____
- ☐ (5) Sample received outside of holding time. _____
- ☐ (6) Sample not properly preserved. pH = ___. Wrong preservative used. _____
- ☐ (7) Illegible sample numbers or label missing from bottles. _____
- ☐ (8) Identification on bottle same as identification on paperwork: yes:_____ no:_____
- ☐ (9) Incomplete instructions received with sample(s). i.e.,
- ☐ no Request for Analysis, no Chain-of-Custody. _____
- ☐ (10) Samples received in improper container. _____
- ☐ (11) Samples held in field before receipt by Lab. Days (specify) _____
- ☐ (12) Air Bubble(s) in ___of ____ samples for volatiles analysis. _____
- ☐ (13) Other _____

CORRECTIVE ACTION: (Check applicable item(s))

		Correction action taken by: Initials	Date
☐	(1) Client informed verbally (Client Services).	_____	
☐	(2) Client informed by memo/letter/fax (Client Services).	_____	_____
☐	(3) Sample processed "as received" (Sample Entry).	_____	_____
☐	(4) Re-sampling requested of client (Client Services).	_____	_____
☐	(5) Samples placed "on hold" until further notice (Sample Entry/Client Services).	_____	_____
☐	(6) NOTE IN NARRATIVE. See temperature/pH login sheet. (Sample Entry).	_____	_____
☐	(7) Other (Specify) _____	_____	_____

* When complete (within 24 hours of nonconformance) forward to QA. Original to be forwarded to initiator to be included in transmittal file.

Comments:

Laucks Testing Laboratories, Inc., Supplemental Sample Receipt Log

Work Order Number: 9601313
Assigned SDG Number: 6527

Temperature		pH or Bottle Types								
		8330								
1	20/23	2)7								

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-4201

Title: **Package Deliverables for all Reporting Levels**

Revision history:

Number	Date
000	1/31/96

Written by: _____ Date: 1-31-96
Tom Marino

Reviewed by: _____ Date: 1-31-96
Charlene Nix

Reviewed by: _____ Date: 1-31-96
Harry Romberg, QC Officer

Approved by: _____ Date: 1/31/96
Karen Kotz, Laboratory Director

UNCON[illegible]D

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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

UNCONTROLLED

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   3.1 Safety Precautions 3
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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.

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

Method #: LTL-7015

Title: **Acid Digestion of Sediments, Sludge, and Soils by SW846 Method 3050B**

Revision history:

Number	Date
0.0	1/18/99
1.0	3/20/00

Revised by: Stan Dittrick — Date: 3/20/00
Stan Dittrick, Chemist

Reviewed by: Paul Swift — Date: 03/21/2000
Paul Swift, Inorganic Division Manager

Reviewed by: Harry Romberg — Date: 3-20-2000
Harry Romberg, QA Officer

Written by: Kathy Kreps — Date: 03-22-2000
Kathy Kreps, Laboratory Director

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1. Introduction and Scope

1.1. This method is an acid digestion procedure used to prepare sediments, sludges, and soil samples for analysis by flame or furnace atomic absorption spectroscopy (FLAA and GFAA, respectively) or by inductively coupled argon plasma spectroscopy (ICP) or inductively coupled argon plasma spectroscopy - mass spectrometry (ICP-MS). Samples prepared by this method may be analyzed by ICP for all the listed metals, or by FLAA or GFAA as indicated below (see also step 2.1):

FLAA/ICP-AES		GFAA/ICP-MS
Aluminum	Magnesium	Arsenic
Antimony	Manganese	Beryllium
*Arsenic	Molybdenum	Cadmium
Barium	Nickel	Chromium
Beryllium	Potassium	Cobalt
Cadmium	*Selenium	Iron
Calcium	Silver	Lead
Chromium	Sodium	Molybdenum
Cobalt	Thallium	Selenium
Copper	*Tin (FLAA only)	*Tin (ICP-MS only)
Iron	Vanadium	Thallium
Lead	Zinc	

* Although Arsenic and Selenium (analyzed by FLAA/ICP), and Tin (analyzed by FLAA or ICP-MS) are not listed in the original SW846 document as analytes that may be analyzed from samples prepared by Method 3050B, Laucks has demonstrated adequate recovery for these analytes from this digestate.

and the Lithium, Strontium and thorium (analyzed by ICP-MS)

Lithium *
Strontium *
Thorium *

HSP
4/20/00

2. Summary of Method

2.1 A representative 1. to 1.5g sample is digested in nitric acid and hydrogen peroxide. The digestate is then either reduced to a low volume or refluxed with hydrochloric acid. Hydrochloric acid is used for flame AA and ICP analyses only. Dilute hydrochloric acid is used as the final reflux acid for (1) the ICP analysis of As and Se, (2) the flame AA or ICP analysis of Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, Pb, Sb, Sn, Tl, V, and Zn. The hydrochloric acid step is omitted for the furnace AA analysis of As, Be, Cd, Cr, Co, Fe, Pb, Mo, Se, and Tl. The diluted samples have an approximate acid concentration of 5% (v/v). add Li, Sr, Th

KSP 4/20

3. Safety Procedures

3.1 These procedures involve the use of strong and/or hot acid solutions. The analyst must wear eye protection, a lab coat, and gloves to protect against burns.

3.2. These procedures involve hot plates which may present the danger of burns from heated surfaces and electrical hazards. The analyst must take appropriate caution to avoid injury from these sources.

3.3. Samples and spiking solutions may contain high levels of toxic metals and other unknown constituents. The analyst must take every precaution to avoid contact with these potentially hazardous materials and should wash hands thoroughly before taking any breaks, eating, or going home for the day.

3.4. Fertilizer samples are chemically complex matrices and should be handled with extreme caution. Many components found in fertilizer are highly reactive to acid and all acid additions should be made with special care, in a hood, standing back from the sample. Also, these samples have been known to "pop" or explode during heating, so care should be taken not to stand close to or have any body part over the samples while they are being heated.

4. Equipment

- 250-mL Erlenmeyer flasks or equivalent, acid-washed
- Analytical balance capable of accurately weighing to the nearest 0.01 g
- Reflux caps fitted for Erlenmeyer flasks, acid-washed
- Eppendorf or other micropipets
- Hot plate or equivalent heating source, adjustable and capable of maintaining a temperature of 90-95° C
- 100-mL graduated cylinder, acid-washed

- 100-mL graduated cylinder, acid-washed
- Filter paper, Whatman No. 41 or equivalent
- Glass funnels, acid-washed
- 100-mL volumetric flasks, acid-washed
- Sample digestate bottles, acid-washed
- Thermometer

Note: All glassware used for this digestion must be prepared by the method stated in Inorganics Glass Cleaning Procedures, SOP #: LTL-7003.

5. Reagents

5.1 Reagent Water. Reagent water will be interference-free deionized water. All references to water in the method refers to reagent water unless otherwise specified.

5.2 Nitric acid (concentrated), HNO_3, ACS Reagent grade or better

5.3 Hydrochloric acid (concentrated), HCl, ACS Reagent grade or better

5.4 Hydrogen peroxide (30%), H_2O_2, ACS Reagent grade or better

The method blank prepared along with the samples is used as a contamination check. Since the holding time for all analytes associated with this method have a holding time of 6 months, repreparation would not be an issue if contamination was traced to a specific reagent.

6. Acid Digestion Procedure

See Appendix A for digestion logs.
See Appendix B for flowchart.
See Appendix C for Quality Control solutions

6.1 Mix the sample thoroughly to achieve homogeneity. For each digestion procedure, weigh 1.00-1.50 g of sample to the nearest 0.01 g directly into an Erlenmeyer flask. For samples with low percent solids, a larger sample size may be used to attain the equivalent of 1.00-1.50 g dry basis as long as digestion is completed. Add appropriate spiking mix aliquots to QC samples at this point.

6.2 Add 5 mL of water and 5 mL of concentrated HNO_3, and mix the slurry, place the watch glasses on the beakers, Heat the samples to 95° C and reflux without boiling for 15 minutes. Record the temperature achieved during the digest on the digest log. Allow the samples to

cool, then add 5 mL of concentrated HNO_3. Heat the sample to reflux for 30 minutes. Repeat this step (addition of 5 mL of concentrated HNO_3) until no brown fumes are given off by the sample indicating the complete reaction with HNO_3. Reduce the volume to 5 mls. Allow the samples to cool, add 2 mL water and 3 mL 30% H_2O_2, and reflux the samples for 10 minutes. Cool the samples, add 3 mL 30% H_2O_2, and reflux the samples until effervescence subsides. Cool, add seven 1 mL aliquots of 30% H_2O_2 separately, and reduce volume to 5 mls. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence after the peroxide additions.

6.3 A. If the sample is being prepared for (a) the ICP analysis of As and/or Se, (b) the flame AA or ICP analysis of Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, Pb, Sb, Sn, Tl, V, and/or Zn, then add 10 mL of concentrated HCl, return the flask to the hot plate, and reflux for an additional 15 minutes without boiling. After cooling, dilute the sample to 100 mL with water in a graduated cylinder and place in an acid-washed digestate bottle. If only one graduated cylinder is to be used to volume all samples, the blank is to be the last sample volumed in order to assure against sample carry over. Navy projects require the use of individual graduated cylinders for each sample.

B. If the sample is being prepared for GFAA analysis of As, Be, Cd, Cr, Co, Fe, Pb, Mo, Se, and/or Tl, the HCl addition is omitted. The sample is diluted to a final volume of 100 mL in a graduated cylinder, and stored in an acid-washed digestate bottle. If only one graduated cylinder is to be used to volume all samples, the blank is to be the last sample volumed in order to assure against sample carry over.

Note: If a sample is allowed to go to dryness at any stage of the digestion procedure, the sample must be discarded and prepared again.

6.4 If silicates or other insoluble material that could clog the nebulizer or the graphite furnace autosampler are present in the samples, the samples should be allowed to settle overnight prior to analysis. Filtration should be performed only if there is concern that the insoluble material will not settle out of solution. Filter and dilute the samples to a final volume of 100 mL, using acid-washed filter apparatus to avoid sample contamination.

6.5 The prepared samples are then transferred to the metals analysis department. The digestion log is to reflect the time at the start and finish of the digest. In order to maintain a strict chain of custody, the time and date when the digestates are relinquished to the analysis department, as well as the initials of the analyst accepting the digestates, are recorded on the digestion log

7. Quality Control

7.1 In each batch or SDG consisting of no more than 20 samples, a preparation blank is created. This consists of an empty, acid-washed Erlenmeyer flask to which the appropriate reagents are added and digested in exactly the same manner as a sample The blank should be labeled on the digestate bottle in the following way: B, date, instrument, S or W (for soil or water) and the sequence number of the digestion. Example: B111794ICPS01.

7.2 Spiked samples should be employed to determine accuracy. In each batch or SDG consisting of no more than 20 samples, either matrix spike / sample duplicate (MS/Dup) or matrix spike / matrix spike duplicate (MS/MSD) samples will be prepared. For SW846-specified quality control measures, MS/Dup samples will be prepared in the following way. Dispense one sample in triplicate. Two will be digested exactly as any other sample. The third must be spiked with the appropriate spike solutions determined by the analyst, and then treated as any other sample. All glassware and digestate bottles must be marked appropriately. If MS/MSD samples are required instead of the SW846-specified MS/Dup, both the second and third aliquots must be spiked and all glassware and digestate bottles marked appropriately. See Appendix C for instructions on the preparation and use of QC solutions. Spiking levels presented are to be used only as guidance. The actual analytes and concentrations may vary.

7.3 Determine the analytes required for analysis by consulting sample work order information. Each spiking solution is given its own unique number according to the page and line of the standards logbook which it is entered into. This number and the volume dispensed must be clearly recorded on the digestion log. A sample page from the standards logbook is included in the Appendix C.

7.4 In each batch or SDG consisting of no more than 20 samples, either a Blank Spike (BLK SPK) or a Laboratory Control Sample (ICP or GF LCSS) must be digested in exactly the same manner as a sample. A Blank Spike consists of an empty, acid-washed Erlenmeyer flask to which the appropriate spikes (determined as outlined in Section 7.3) and reagents are added. The Certified Value and Advisory range for each analyte in a particular LCSS can be accessed from the Quality Control Database.

8. References

8.1 USEPA, Test Methods for Evaluating Solid Waste Physical/Chemical Methods, USEPA SW846, most recent version, Method 3050B.

Appendix A. LTL-7015 Digestion Logs

LAUCKS TESTING LABORATORIES, INC.					GRAPHITE FURNACE DIGESTION LOG				Place a √ in the box next to the method performed (circle version)			
SDG #:	Digest start : date/time				Digest finish : date/time				SW-846 3020A		CLP-water	
Analyst:	/				/				SW-846 3050A/B		CLP-soil	
Sample ID	Sample Size (g:mL)	Final Volume (mL)	Color		Clarity		Text	Art	Comments			
			Bef	Aft	Bef	Aft						

Chain of Custody	Digest Relinquished Date / Time : Analyst	Digest Accepted Analyst	Digest Disposed Date	Hot Plate Temperature, °C
	/			

Color 1=Red, 2=Blue, 3=Yellow, 4=Green, 5=Orange, 6=Violet, 7=White, 8=Brown, 9=Grey, 10=Black, 11=Colorless
Clarity 1=Clear, 2=Cloudy, 3=Opaque
Texture F=Fine (powdery), M=Medium (sandy), C=Coarse (rocky) Artifacts: If "Yes", give description in the comments field.

P:\METALS\BNCHSHE\DIG.D

LAUCKS TESTING LABORATORIES, INC.			ICP DIGESTION LOG		Place a √ in the box next to the method performed (circle version).			
SDG #:	Digest start : date/time		Digest finish : date/time		SW-846 3010		CLP-water	
Analyst:	/		/		SW-846 3050A/B		CLP-soil	

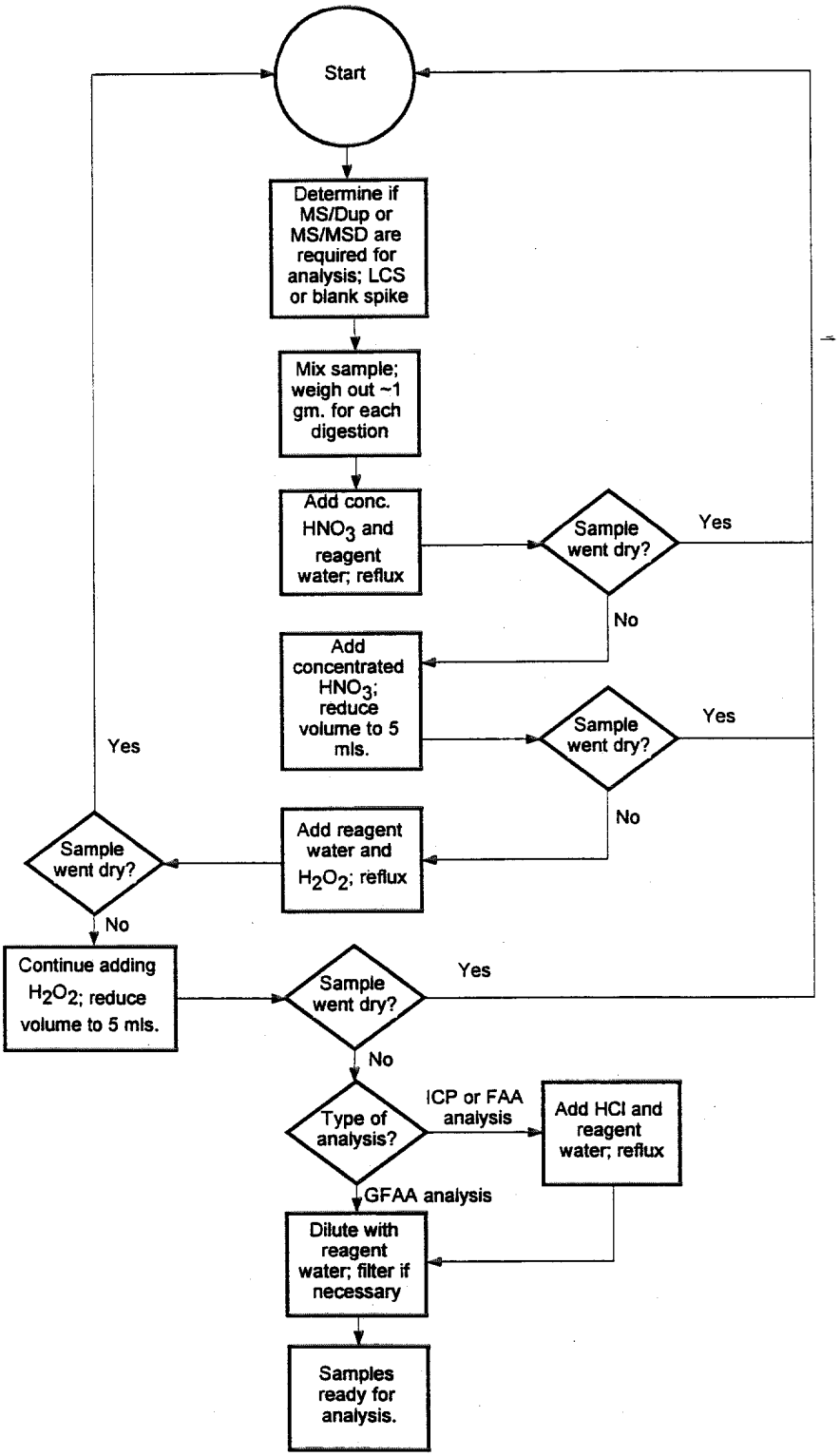
Sample ID	Sample Size (g:mL)	Final Volume (mL)	Color		Clarity		Text	Art	Comments
			Bef	Aft	Bef	Aft			

Chain of Custody	Digest Relinquished Date / Time : Analyst	Digest Accepted Analyst	Digest Disposed Date	Hot Plate Temperature, °C
	/			

Color 1=Red, 2=Blue, 3=Yellow, 4=Green, 5=Orange, 6=Violet, 7=White, 8=Brown, 9=Grey, 10=Black, 11=Colorless
Clarity 1=Clear, 2=Cloudy, 3=Opaque
Texture F=Fine (powdery), M=Medium (sandy), C=Coarse (rocky) Artifacts: If "Yes", give description in the comments field.

P:\METALS\BNCHSHEEPDIG.D

Appendix B, Flowchart for LTL-7015


Start
Determine if MS/Dup or MS/MSD are required for analysis; LCS or blank spike
Mix sample; weigh out ~1 gm. for each digestion
Add conc. HNO_3 and reagent water; reflux
Sample went dry?
Yes
No
Add concentrated HNO_3; reduce volume to 5 mls.
Sample went dry?
Yes
No
Add reagent water and H_2O_2; reflux
Sample went dry?
Yes
No
Continue adding H_2O_2; reduce volume to 5 mls.
Sample went dry?
Yes
No
Type of analysis?
ICP or FAA analysis
Add HCl and reagent water; reflux
GFAA analysis
Dilute with reagent water; filter if necessary
Samples ready for analysis.

Appendix C, Quality Control Solutions

ICP Spikes

- The CLP spike solution for ICP digest was made by diluting 10 mLs of CLPP-SPK-1™, 10 mLs of CLPP-SPK-2™, and 10 mLs of CLPP-SPK-3™ to 100 mLs with Type II water.

- Add 1000 µLs of CLP spike solution to water samples and 1000 µLs of CLP spike solution to soil samples prior to digestion.

ICP Analytes

Analyte	Concentration in Digest (ppb)	Analyte	Concentration in Digest (ppb)
Ag	50	Fe	1000
Al	2000	Mn	500
As	2000	Ni	500
Ba	2000	Pb	500
Be	50	Se	2000
Cd	50	Sb	500
Co	500	Tl	2000
Cr	200	V	500
Cu	250	Zn	500

Table based on a final volume of 100 mL for water digests, and 100 mL for soil digests.

GRAPHITE FURNACE SPIKES

- Add the analytes listed in the table below to a 100 mL volumetric flask which contains ~50 mL of Type II water and 5 mL of HNO_3.

Analyte	Volume	Stock Concentration
As	400 µL	1000 ppm
Pb	200 µL	1000 ppm
Se	100 µL	1000 ppm
Tl	500 µL	1000 ppm

- Dilute to 100 mL with Type II water. This spike solution now contains:

4.0 ppm	Arsenic
2.0 ppm	Lead
1.0 ppm	Selenium
5.0 ppm	Thallium

- Add 1000 µL of Spike Solution to water samples and 1000 µL to soil samples prior to digestion.

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-7105

Title: Method Protocols for the Analysis of Metals by ICP Using SW 846 6010B

Revision history:

Number	Date
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UNCONTROLLED

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1. Introduction and Scope

1.1 Scope

This method is to be used as a supplement to the instrumental SOP in order to follow the method requirements of SW 846 6010B for ICP analysis. Operating parameters are to be followed in the individual instrument SOP.

The analyst should become familiar with SW 846 protocols prior to using this SOP.

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

Samples are to be collected in either glass or plastic containers. Water samples are to be preserved to a pH < 2. A one liter sample bottle is sufficient volume for analysis. Soil samples do not require preservation but need to be stored at 4° C. At least 200 grams of sample should be collected. The holding time for ICP metals is 6 months. See Appendix III for Sample Handling and Preservation Table.

1.3 Definition of Terms

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

ICV - Initial Calibration verification - This is a standard run immediately following the initial calibration. The ICV is made from an independent source. Agreement within 10% and a relative standard deviation less than 5% RSD from replicate (minimum of two) integrations, is required.

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

CCV - Continuing calibration verification. - This is a standard analyzed after every 10 samples during the analysis sequence to determine whether the instrument or system has

remained in calibration. Agreement within 10% and less than 5% RSD from replicate (minimum of two) integrations, is required.

CCB - Continuing Calibration Blank - This is a blank that is used to determine if there is carry-over between sample injections. A CCB succeeds every CCV, every 10 samples.

ICSA - Interference Check Solution A - This solution is prepared to contain known concentrations of interfering elements that will provide an adequate test of the correction factors.

ICSAB - Interference Check Solution AB - This solution is prepared by spiking the ICSA with known quantities of analyte. Adequate recovery of the analytes within this interfering matrix indicates proper application of the correction factors.

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

IDL - Instrument detection limit - The lowest concentration of a target analyte that is detectable. The IDL is three times the average standard deviation of seven replicates at 2 to 5 times the estimated IDL over three non consecutive days. Used as a starting point for selecting MDL study spiking levels. IDL should be determined quarterly.

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

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

QC period - Quality control period - An analysis sequence initiated by the analysis of one or more standards, followed by samples, and terminated with a standard and blank analysis.

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

Sequence - A set of sample digests and standard solutions introduced into an instrument in a chronologically continuous group.

2. Equipment List and Standards

2.1 Instrumentation:

Thermo Jarrell Ash Enviro 36 simultaneous ICP or equivalent.

2.2 Standards

SW 846 requires the use of one standard and a blank. See the instrument SOP for standards and their preparation.

3. Safety precautions and Waste Disposal

See instrument SOP for detailed safety precautions and waste disposal.

Safety

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

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

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

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

Caution should be used when handling acidic digestates.

3.1 Waste Disposal

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

Prior to the analysis of any samples, it is necessary to establish method detection limits. This procedure is fully described in the Laucks SOP on performing MDL studies. Briefly, it involves the analysis of 7 replicate samples spiked at a concentration approximately 3 to 5 times the estimated method detection limit. A Student's T-test is then applied to these measured values to calculate the MDL.

4.2 Linear range study

The upper limit of the linear dynamic range must be established by running a multi-point calibration using 4 standards and then running an upper range standard. The upper range limit should be an observed signal with no more than a 10% variation from the true value of the upper range standard. All samples with elements exceeding the level of the upper range standard must be diluted.

4.3 Initial Calibration

Analyze standard solutions using a minimum of a calibration blank and one standard. The calibration curve must be verified by running an Initial Calibration Standard (ICV) and obtaining agreement within 10% of the expected concentration and a 5% RSD for replicate integrations.

Criteria and Corrective Action:

Since a linear regression is not possible when using a two point calibration on the Enviro 36, the standard curve is validated by evaluating the ICV and the subsequent CCVs. If the corresponding control limits for the ICV and CCV are exceeded, then the sample analysis must be discontinued, the cause determined and the instrument recalibrated. All samples following the last acceptable ICV, CCV must be reanalyzed..

4.4 Initial Calibration Standard ICV:

Immediately after the calibration curve, an ICV is analyzed.Criteria:

The calculated concentration of the standard must be within 10% of the expected value and the RSD must be less than 5% for replicate integrations.

Corrective Action:

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

4.5 Initial Calibration Blank

After the analysis of the high standard, an instrument blank (ICB) is analyzed. The levels of target analytes in the ICB should not exceed three times the instrument detection limit.

Corrective Action:

If the ICB analyte levels exceed 3 times the IDL, repeat the analysis two more times and average the results. If the average is not within three standard deviations of the backgroundl mean, the system is out of control. The source of contamination must be identified and corrected before proceeding with the analysis.

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

A continuing calibration verification standard is analyzed after every 10 samples. Immediately following the CCV, a blank solution is analyzed. In addition, this standard and blank must be the last samples analyzed in the run. The CCV must also be prepared from an independent source.

The CCV must fall within ± 10% of the true value and the RSD must be less than 5% for replicate integrations.

The levels of target analytes in the CCB should not exceed 3 times the IDL.

Corrective action:

If CCV limits are exceeded, check calculations or perform instrument maintenance. Recalibrate and reanalyze. No sample results may be reported that are not bracketed by a

successful calibration and a CCV/CCB which is in control or by preceding and following CCV/CCBs which are within limits.

If the CCB analyte levels exceed 3 times the IDL, repeat the analysis two more times and average the results. If the average is not within three standard deviations of the background mean, terminate the analysis, correct the problem, recalibrate, and reanalyze the previous 10 samples. If the blank is less than 1/10 the concentration of the action level of interest, and no sample is within ten percent of the action limit, analyses need not be rerun and recalibration need not be preformed before continuation of the run.

4.7 Interference Check Solutions A (ICSA) and AB (ICSAB)

(ICSA):

At the beginning and at the end of each run, an interference check solution A is analyzed. This solution contains interefering elements only. All other elements are not present in the solution. All elements not present should show a recovery of zero, or ± the CRDL.

Corrective Action:
If the analytes do not recover within the specified control limits, then the system is out of control. The problem needs to be identified and corrected prior to beginning another run.

(ICSAB):

At the beginning and end of each analytical sequence an ICSAB must be analyzed. Analytes must recover between 80-120%.

Corrective Action:

If the analytes do not recover within the specified control limits, then the system is out of control. The problem needs to be identified and corrected prior to beginning another analysis.

4.8 Method Blanks

Method blanks are used to verify contamination free reagents and apparatus. They are prepared with every set of samples prepared at the same time or at least one blank every 20 samples, which ever is more frequent. Any analyte response above the MDL is reported. For a method blank to be acceptable for use with the accompanying samples, the concentration of the blank of any analyte of concern should not be higher than the highest of either:

(1) The reporting limit, or
(2) Five percent of the regulatory limit for that analyte, or
(3) Five percent of the measured concentration in the sample.

Corrective Action:

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

4.9 Laboratory Control Sample

The LCS is made from an independent source of the same matrix (soil or water) and is carried through the entire digestion procedure. An LCS is performed with each digestion batch. At a minimum, LCSW(water) control limit are 80% to 120%. Control limits for the LCSW will be empirically determined and must be within the method specified control limit noted aboved.

LCSS(soil) control limits are supplied by the manufacturer. LCSS control limits are not derived by the laboratory due to the small number of data points available from each lot of certified material.

Corrective Action

If the LCS is not within the required control limits, then a redigestion will occur for the affected analytes.

4.10 Matrix Spike

A sample is chosen at random from the samples to be analyzed, and an aliquot of spiking solution is added to this sample prior to preparation. The analyst should attempt to avoid selecting samples which are identified by the client as blanks. As the purpose of the matrix spike is to test the system under "typical" conditions, the analyst may also avoid selecting

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

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

where:

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

For ICP, control limits for spike recoveries will be 75-125% unless otherwise specified in the project specific QAPP. In-house control limits are based on historical performance. The recovery criteria are detailed in the QC Database QC_DB and will change from time to time.

Corrective Action:

Samples with spike recoveries outside control limits will be reviewed for possible corrective action. Corrective action will first involve recalculation, followed by possible re-preparation, and/or reanalysis. This process should also look at the recovery of matrix spiking compounds from the SRM and/or blank spike analysis. In some cases a Post Digestion Spike is required when matrix interference is suspected. In all cases a narrative explanation of the condition is required to detail the corrective actions taken. Data reported in validatable packages will be flagged with an "N" indicating the out-of-control event..

4.11 Matrix Spike Duplicate/Sample Duplicate

Method QC consists of MS/MSD. A duplicate maybe be performed instead of a MSD. Other types of QC can performed at the client's request.

Criteria

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

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

where:
SS = concentration in spiked sample
SSD = concentration in matrix spiked duplicate sample

For sample concentractions greater than ten times the IDL, control limits for RPD of duplicates will be ±20% unless otherwise specified in the project specific QAPP. For sample concentrations less than ten times the IDL, control limits for the difference will be ± the reporting limit. In-house control limits are based on historical performance. The RPD control limits are detailed in the current QC Database QC_DB and will change from time to time.

Corrective Action:

If a trend in out of control RPD values is observed, the methods used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable reproducibility. Generally, if recoveries are in control and no analyte of interest was detected in any of the samples, no immediate action will be taken on that sample set. If integrity of reported sample values is in doubt, re-analysis may be called for. Corrective actions should be discussed with the Quality Control Officer. In a validatable package, data associated with an out-of-control RPD will be flagged with an "*".

4.12 Serial Dilution

A five-fold dilution is performed on the QC sample in each analytical batch. The difference between the initial value and the serial dilution should agree within 10%. If the difference is greater than 10% on analytes that exhibit a level 50 times greater than the MDL, then results for those analytes will be flagged with an "E".

Corrective Action:

No corrective action is necessary other than appropriately flagging the data.

5. Operation procedures

- Calibrate the instrument with a blank and one standard.

- Immediately following the calibration, analyze the ICV followed by an ICB. Concentration values obtained for the ICV should not deviate from the actual values by more than 10% and less than 5% RSD for replicate integrations.
- Flush the system with the calibration blank solution for at least 1 minute before the analysis of each sample.
- Analyze a CCV and CCB every 10 samples. The CCV must agree within 10% of its expected value and less than 5% RSD for replicate integrations. The results of the calibration blanks are to be within 3 times the IDL. If not, repeat the analysis two or more times and average the results. If the average is not within 3 times the IDL, terminate the analysis, correct the problem, recalibrate, and reanalyze the previous 10 samples.

5.1 Analysis Sequence

See Appendix IV for Analytical Sequence

5.2 Instrumental Conditions

See instrument SOP for operating procedures.

5.3 Analytical Operation

See instrument SOP for operating procedures.

6. Reports

6.1 Data Packet Organization

- See the SOP metals validation for a check list detailing data packet organization
- If requested, all analysis performed under SW 846 guidelines can be reported via CLP SOW 3/90 forms. These forms provide all relevant QC information.
- Data packages will be produced via Enviroforms. Analyte levels that are less than the MDL will be reported as the SDL followed by a "U". Analyte levels that fall between the MDL and the reporting limit will be flagged with a "B". Analyte levels greater than or equal to the reporting limit PQL will be reported without a flag.

CODE	Definition	
U	The analyte of interest was not detected, to the limit of detection indicated.	❁◆
B	The analyte of interest was detected between the MDL and the reporting limit.	❁
N	The spike recovery exceeded the control limits.	❁
*	The duplicates exceeded the RPD control limit or their difference exceeded the reporting limit.	❁
E	The Serial Dilution did not agree within 10%.	❁
S	The analyte concentration was determined by MSA.	❁

◆ Used in all reports.
❁ Used in data validatable packages.

6.2 Control Chart(s)

The recovery values for ICP analytes Al, B, Cd, Cr, and Ag in the LCS are plotted on control charts. Other analytes may be added at the discretion of QA without immediate revision of this SOP.

6.3 References:

Test Methods for Evaluating Solid Waste, SW-846, Method 6010B, Revision 2, December 1996

Appendix I

QC Summary Table

Laucks Testing Laboratories

Method 6010B QA Requirements and Corrective Actions

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Initial Calibration	One standard and a Blank	One standard and a Blank	Beginning of each run.		In raw data.
Initial Calibration Verification	±10% of nominal value. Made from an independant source.	±10% of nominal value. Made from an independant source.	Beginning of each run immediately following ICAL.	Recalibration required.	Form 2A or in raw data.
Continuing Calibration Verification	±10% and RSD < 5% for replicate integrations.	±10% and RSD < 5% for replicate integrations.	Every 10 samples. Mid-range	Recalibrate and rerun affected samples.	Form 2A or in raw data.
Instrument Blank	±3 sigma of average.	±3 sigma of average	Every 10 samples.	Recalibrate and rerun affected samples.	Form 3 or in raw data.
Method Blank	< MDL	< reporting limit	One/batch	Redigest samples	Form 3, in raw data, or database report.
Laboratory Control Sample		LCSW: 80%-120% LCSS: Manufacturer Specs.	One/batch	Redigest samples	Form 7, in raw data, or database report
Matrix Spike Recovery	75-125%	75-125% or current QC database criteria.	5% or per batch	Consult QCO	Form 5, in raw data, or database report
Duplicate % Difference	± 20%	± 20% or current QC database criteria.	5% or per batch	Consult QCO	Form 6, in raw data, or database report
Serial Dilution	± 10% difference	± 10% difference	One/batch	Flag data with an "E".	Form 9 or in th raw data.
ICSA,AB	±20% true value of analytes, or ± the CRDL.	±20% true value of analytes, or ± the CRDL.	Beginning and end of run	Consult QCO	Form 4, or in raw data.

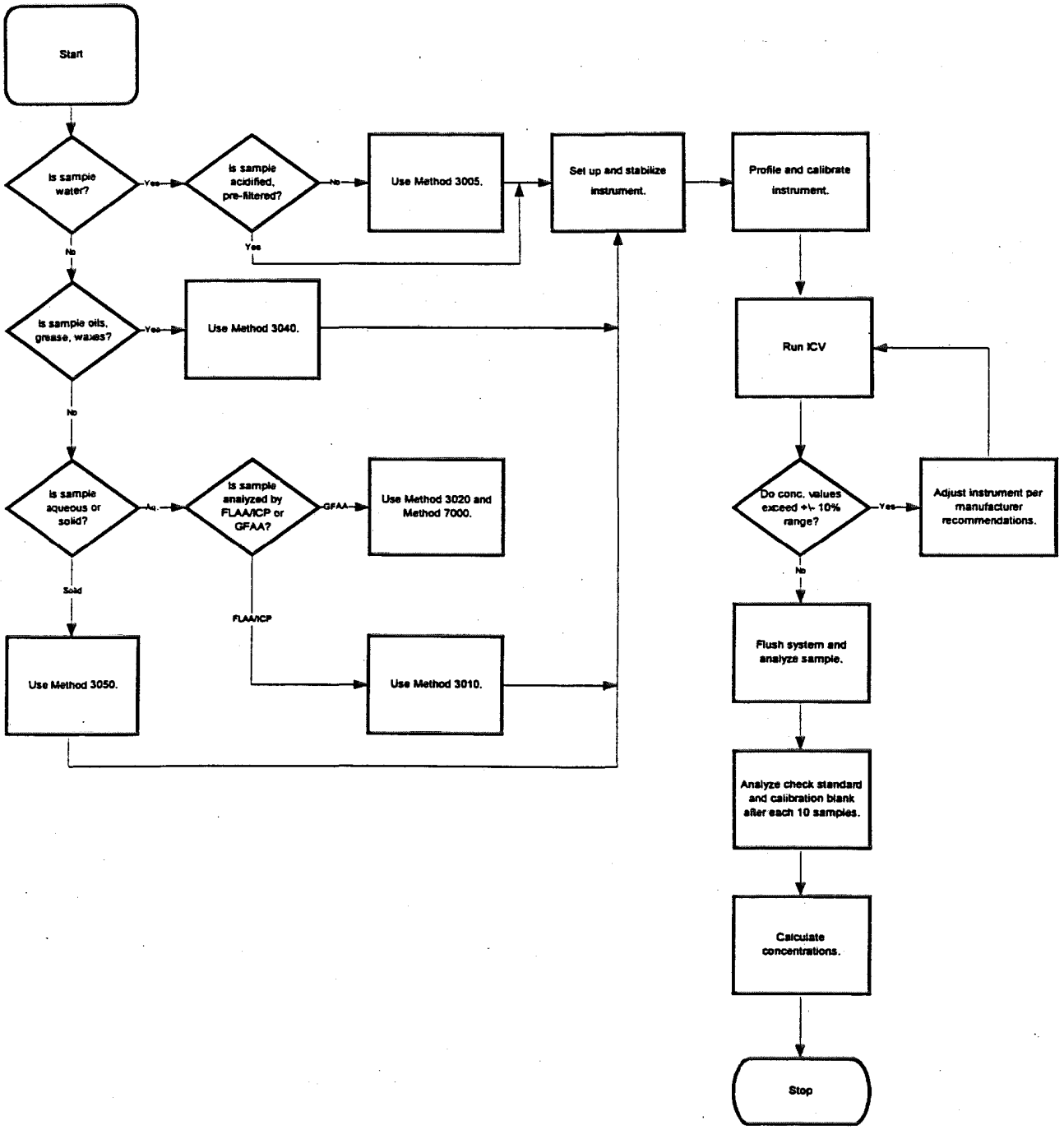
Laucks Testing Laboratories

Method 6010B QA Requirements and Corrective Actions

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentatic
Standard Reference Material (SRM) Recovery	See QC control catalog	Control limits set by vendor	5% or per batch	Redigest samples	Form 7, in raw data, or database repor

Appendix II

Flow Chart


Start
Is sample water?
Yes
Is sample acidified, pre-filtered?
No
Use Method 3005.
Yes
Set up and stabilize instrument.
Profile and calibrate instrument.
No
Is sample oils, grease, waxes?
Yes
Use Method 3040.
Run ICV
No
Is sample aqueous or solid?
Aq.
Is sample analyzed by FLAA/ICP or GFAA?
GFAA
Use Method 3020 and Method 7000.
Do conc. values exceed +\- 10% range?
Yes
Adjust instrument per manufacturer recommendations.
No
Solid
FLAA/ICP
Use Method 3050.
Use Method 3010.
Flush system and analyze sample.
Analyze check standard and calibration blank after each 10 samples.
Calculate concentrations.
Stop

Appendix III

Sample Handling and Preservation

Metals (except hexavalent chromium and mercury):

Measurement	Digestion Volume Required.	Collection Volume	Preservation/Holding Time
Total recoverable	100 mL	600 mL	HNO_3 to pH <2 6 months
Dissolved	100 mL	600 mL	Filter on site; HNO_3 to pH <2 6 months
Suspended	100 mL	600 mL	Filter on site 6 months
Total	100 mL	600 mL	HNO_3 to pH <2 6 months

Solid samples should be at least 200 g and usually require no preservation other than storing at 4°C until analyzed. Either plastic or glass containers may be used for sample collection.

Appendix IV

Analytical Sequence

Standard 0
Standard 1
ICV
ICB
CCV
CCB
ICSAI
ICSABI
Sample #1
Sample #1D
Sample #1S
Sample #1L
Sample #2
Sample #3
Sample #4
ICSAF
ICSABF
CCV
CCB

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-7202

Title: Metals Analysis Using Inductively Coupled Plasma - Mass Spectrometry (ICP/MS), SW 846 Method 6020

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Written by: _____ Date: 7-22-99
Ming-Hwa Hwang, Metals Supervisor

Approved by: Harry Romberg Date: 7-22-99
Harry Romberg, QA Officer

Approved by: Kathy Kreps Date: 7-22-99
Kathy Kreps, Laboratory Director

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1. Introduction and Scope

1.1 Method Description

Inductively coupled plasma-mass spectrometry (ICP-MS) is a technique which is applicable to µg/L concentrations of a large number of elements in water and wastes after appropriate sample preparation steps are taken. When dissolved constituents are required, samples must be filtered and acid preserved prior to analysis. No further digestion is required prior to analysis for dissolved elements. Acid digestion prior to filtration and analysis is required for groundwater, aqueous samples, industrial waste, soil, sludge, sediment, and other solid waste for which total (acid-leachable) elements are required.

See Appendix V for reporting limits.
See Appendix I for analytical masses and standard concentrations.

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

1.2 Sample Collection, Sample Storage, Holding Times

Water samples should to be collected in plastic or Teflon containers and preserved to a pH < 2. A one liter sample bottle is sufficient volume for analysis. Soil samples do not require preservation but need to be stored at 4° C and may be collected in glass if plastic containers are not available. At least 200 grams of sample should be collected. The holding time for metals is 6 months. If mercury is being analyzed by this technique, which is not currently approved or done without specific client arrangement, the holding time is 28 days.

1.3 Definition of Terms

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

Batch Identifier - A number given to each analysis group which uniquely identifies that batch. This number is preceded by an "A", mmddyy, ICPMS, matrix (W for water, S for soil), sequence number (i.e. A022595ICPMSW01).

CCB - Continuing Calibration Blank - This is the same acronym used in the CLP program. This is a blank which is analyzed immediately after the CCV (almost always after every 10 samples and at the end of the analytical run) during the analysis sequence to determine whether the instrument or system has maintained a stable baseline.

CCV - Continuing calibration verification. - This is the same acronym used in the CLP program. This is a standard analyzed at some prescribed frequency (almost always after every 10 samples and at the end of the analytical run) during the analysis sequence to determine whether the instrument or system has remained in calibration.

CLP - Contract Laboratory Program - The USEPA program that contracts with laboratories to provide laboratory services. The term has come to mean a much broader set of methods and deliverables. In context of this SOP, CLP means procedures or operations which are detailed in the CLP contract and which are extended to a broader working definition.

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

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

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

IDL - Instrument detection limit. IDL's can be estimated by analyzing seven replicates of a standard analyte solution over three nonconsecutive days. The analyte concentration should be 3-5 times the estimated IDL. Multiplying the average standard deviation by three will yield the IDL for that analyte. Each measurement must be performed as though it were a separate analytical sample. IDL's must be determined quarterly

MDL - Method detection limit - The lowest concentration of an analyte which will yield a positive result that is greater than zero at a known level of confidence. MDLs are empirically determined and are performed annually.

LCS - Laboratory Control Sample. This is a material of approximately the same matrix as the samples, containing a known and usually certified amount of target analyte and which is prepared and analyzed in the same manner as a typical sample. This

sample is used to demonstrate that the analytical system is in control. It may be considered to be a blank spike for most inorganic analyses and is preferred over artificially spiking blank materials.

Serial Dilution - If the analyte concentration is within the linear range of the instrument and is sufficiently high (minimally, a factor of 100 above the IDL/MDL), an analysis of a fivefold dilution must agree within ±10% of the original determination. If not, an interference effect must be suspected. One serial dilution must be analyzed for each twenty samples or less of each matrix in a batch. A serial dilution is denoted in the raw data by an "L".

ICSA - Interference Check Solution A. The ICSA is a solution that contains the interfering analytes. This solution is analyzed to indicate if a high level of interfering compounds will have an affect on the analytes of interest.

ICSAB - Interference Check Solution AB. The ICSAB is a solution that contains the interfering analytes and the analytes of interest. This solution is analyzed to indicate if a high level of interfering compounds will have an affect on the recovery of the analytes of interest.

Internal Standards - Internal standards are added to all blanks, standards, and samples. They monitor the affect of a sample's matrix on the quantification of the analytes of interest. The internal standards used are Sc45, In115, and Bi209.

Post-Digestion Spike - An analyte spike added to a portion of a prepared sampleshould be recovered to within 75% to 125% of the known value or within the laboratory derived acceptance criteria.

Standard-Addition - The standard addition technique involves adding known amounts of standard to an aliquot of the sample. This technique compensates for a sample consitituent that enhances or depresses the analyte signal thus producing a different slope than that of the calibration standards.

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

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

2. Equipment List and Standards

2.1 Instrumentation:

Perkin-Elmer ELAN 5000

2.2 Standards

SW 846 requires the use of one standard and a blank. Standards are made in a 1% HNO_3. See Appendix I for standard concentrations.

2.3 Internal Standards

Sc45, In115 and Bi209 are used as internal standards.

3. Safety precautions and Waste Disposal

3.1 Safety Precautions

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

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

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

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

Radio Frequency - The ELAN 5000 uses a high energy RF. Although the generator is well shielded, care should be taken when operating the instrument. Pace makers can be adversely affected by exposure to high energy RF.

Plasma Radiation - Do not view the torch without the proper eye wear. Severe eye damage can occur if the plasma is viewed directly.

3.2 Waste Disposal

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

4. Quality Control

4.1 Method Detection Limit Study

Prior to the analysis of any samples, it is necessary to establish method detection limits. This procedure is fully described in the Laucks SOP on performing MDL studies. Briefly, it involves the analysis of 7 replicate samples spiked at a concentration approximately 3 to 5 times the estimated method detection limit. A Student's T-test is then applied to these measured values to calculate the MDL.

4.2 Linear range study

Linear ranges for each analyte are determined by analyzing a high concentration "sample". The analytically determined concentration must be within 5% of the true value. The true value is the upper limit of the ICP/MS linear range. Linear ranges must be verified quarterly.

4.3 Internal Standards

A 50 uL aliquot of a 10 ppm stock solution of Sc45, In115 and Bi 209 is added to a 10 mL of all standards and samples prior to analysis.

Criteria

The intensities of all internal standards for instrument check standards must be between 80 and 120 percent of the intensities of the internal standards in the initial calibration standard.

Corrective action

If the criteria are not met, terminate the analysis, correct the problem, recalibrate, verify the new calibration, and re-analyze the affected samples.

Criteria

The intensities of all internal standards in the samples must be between 30 and 120 percent of the intensities of the internal standards in the initial calibration standard.

Corrective action

When the intensity of any internal standard in the sample fails to fall between the required levels, the sample must be diluted fivefold (1+4) and re-analyzed with the addition of appropriate amounts of internal standards. This procedure must be repeated until all internal standards fall within the prescribed windows.

4.4 Initial Calibration

Analyze standard solutions using a minimum of a calibration blank and one standard. The calibration curve must be verified by running an Initial Calibration Standard (ICV) and obtaining agreement within 10% of the expected concentration.

Criteria and Corrective Action:

Since a linear regression is not possible when using a two point calibration on the ELAN 5000, the standard curve is validated by evaluating the ICV and the subsequent CCVs. If the corresponding control limits for the ICV and CCV are exceeded, then the sample analysis must be discontinued, the cause determined and the instrument recalibrated. All samples following the last acceptable ICV, CCV must be reanalyzed..

4.5 Initial Calibration Verification

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

Criteria

The calculated concentration of the ICV should be within 90%-110% of the true value.

Corrective action

If the ICV criteria are not met, the analysis is terminated. Perform system maintenance and re-calibrate the instrument.

4.6 Initial Calibration Blank

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

Corrective action

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

4.7 Continuing Calibration Verification (CCV) and Blank (CCB)

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

Criteria

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

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

Corrective action

If CCV limits are exceeded, check calculations or perform instrument maintenance. Recalibrate and reanalyze. No sample results may be reported that are not bracketed by a successful calibration and a CCV which is in control or by preceding and following CCVs which are within limits.

If the initial CCB contains target analyte levels above the contract required detection limit, the system is out of control. The source of contamination must be identified and corrected and the affected samples re-analyzed. As with the CCVs, no sample results may be reported that are not bracketed by a successful initial and continuing calibration blank which are in control or by preceding and following CCBs which are within limits.

4.8 Interference Check Solutions A (ICSA) and AB (ICSAB)

Due to the high sensitivity of the ICP-MS technique and instrument developments that have occurred since the method was written, the high dissolved solids content of the specified ICS

solutions are not recommended by the manufacturer for modern instruments. The ICSA and ICSAB solutions are prepared at different concentration levels from method 6020 to avoid clogging of the sampler cone orifice and damage to the instrument. Therefore, Al, Ca, Fe, Mg, Na, P, K, S, C, and Cl in the ICSA and the ICSAB are at 1/10 of the specified levels. See Appendix II for ICSA and ICSAB solutions concentrations.

(ICSA):

At the beginning and at the end of each run, an interference check solution A is analyzed. This solution contains interefering elements only. All other elements are not present in the solution. All elements not present should show a recovery of zero, or ± the contract required detection limit.

Corrective Action:
If the analytes do not recover within the specified control limits, then the system is out of control. The problem needs to be identified and corrected prior to beginning another run.

(ICSAB):

At the beginning and end of each analytical sequence an ICSAB must be analyzed. Analytes must recover between 80-120%.

Corrective Action:

If the analytes do not recover within the specified control limits, then the system is out of control. The problem needs to be identified and corrected prior to beginning another analysis.

4.9 Method Blanks

Method blanks are used to verify contamination free reagents and apparatus. They are prepared with every set of samples prepared at the same time or at least one blank every 20 samples, which ever is more frequent. Any analyte response above the CRDL is reported. For a method blank to be acceptable for use with the accompanying samples, the concentration of the blank of any analyte of concern should not be higher than the highest of either:

(1) The reporting limit, or
(2) Five percent of the regulatory limit for that analyte, or
(3) Five percent of the measured concentration in the sample.

Corrective Action:

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

4.10 Laboratory Control Sample

The LCS is made from an independent source of the same matrix (soil or water) and is carried through the entire digestion procedure. An LCS is performed with each digestion batch. At a minimum, LCSW(water) control limits are 80% to 120%.

LCSS(soil) control limits are supplied by the manufacturer. LCSS control limits are not derived by the laboratory due to the small number of data points available from each lot of certified material.

Corrective Action

If the LCS is not within the required control limits, a redigestion will occur for the affected analytes.

4.11 Matrix Spike

A sample is chosen at random from the samples to be analyzed, and an aliquot of spiking solution is added to this sample prior to preparation. The analyst should attempt to avoid selecting samples which are identified by the client as blanks. As the purpose of the matrix spike is to test the system under "typical" conditions, the analyst may also avoid selecting the most difficult sample of the batch for spiking. The minimum frequency for MS analysis is 1 each per 20 samples per matrix. This will be best accomplished by running one with every batch for many analyses. This matrix spike sample is used to evaluate the matrix effect of the sample upon recovery of the analytes. The recovery of spike analytes is calculated as follows:

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

where:

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

For ICP/MS, control limits for spike recoveries will be 75-125% unless otherwise specified in the project specific QAPP. In-house control limits are based on historical performance. The recovery criteria are detailed in the QC Database QC_DB and will change from time to time.

Corrective Action:

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

4.12 Post-Digestion Spike

A post digestion spike is also performed to a portion of a prepared sample. The minimum frequency for MS analysis is 1 each per 20 samples per matrix, control limits for post-spike recoveries will be 75-125%

Corrective Action:

Samples with post-spike recoveries outside control limits will be diluted and re-analyzed to compensate for matrix effects. The results must agree to within 10% of the origianal measured concentrations. A standard-addition technique may also be used to compensate for matrix effects.

4.13 Matrix Spike Duplicate/Sample Duplicate

Method QC consists of MS/MSD. A duplicate maybe be performed instead of a MSD. Other types of QC can performed at the client's request.

Criteria

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

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

where:

SS = concentration in spiked sample
SSD = concentration in matrix spiked duplicate sample

For sample concentractions greater than 100 times the IDL, control limits for RPD of duplicates will be ±20% unless otherwise specified in the project specific QAPP. For sample concentrations less than 100 times the IDL, control limits for the difference will be ± the reporting limit. In-house control limits are based on historical performance. The RPD control limits are detailed in the current QC Database QC_DB and will change from time to time.

Corrective Action:

If a trend in out of control RPD values is observed, the methods used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable reproducibility. Generally, if recoveries are in control and no analyte of interest was detected in any of the samples, no immediate action will be taken on that sample set. If integrity of reported sample values is in doubt, re-analysis may be called for. Corrective actions should be discussed with the Quality Control Officer. In a validatable package, data associated with an out-of-control RPD will be flagged with an "*".

4.14 Serial Dilution

A five-fold dilution is performed on the QC sample in each analytical batch. The difference between the initial value and the serial dilution should agree within 10%. If the difference is greater than 10% on analytes that exhibit a level 100 times greater than the IDL, then results for those analytes will be flagged with an "E".

Corrective Action:

No corrective is necessary other than appropriately flagging the data.

6. Reports

6.1 Data Packet Organization

- See the SOP metals validation for a check list detailing data packet organization
- If requested, all analysis performed under SW 846 guidelines the data can be reported via CLP SOW 3/90 forms.
- Data packages will be produced via Enviroforms. Analyte levels that are less than the MDL will be reported as the SDL followed by a "U". Analyte levels that fall between the MDL and the reporting limit will be flagged with a "B". Analyte levels greater than or equal to the reporting limit PQL will be reported without a flag.

CODE	Definition	
U	The analyte of interest was not detected, to the limit of detection indicated.	❁◆
B	The analyte of interest was detected between the MDL and the reporting limit.	❁
N	The spike recovery exceeded the control limits.	❁
*	The duplicates exceeded the RPD control limit or their difference exceeded the reporting limit.	❁
E	The Serial Dilution did not agree within 10%.	❁
S	The analyte concentration was determined by MSA.	❁

◆ Used in all reports.
❁ Used in data validatable packages.

6.2 References:

Test Methods for Evaluating Solid Waste, SW-846, Method 6020, Revision 0, September 1994

5. Procedure

5.1 Instrumental Conditions

- Refer to manufacture's instruction for specific operating procedures. Allow at least 30 minutes for the instrument to stabilize before initiating any analysis.

- Conduct mass calibration and resolution checks in the mass regions of interest. The mass calibration and resolution parameters are required criteria which must be met prior to any sample being analyzed. The monitored masses of Mg, Rh, and Pb must meet the following criteria:

Element	RSD for replicate (minimum of four) integrations.	Mass, amu	Resolution @ 10 % peak height, amu
Mg	< 5%	23.90-24.10	<0.9
Rh	< 5%	102.80-103.00	<0.9
Pb	< 5%	207.90-208.10	<0.9

5.2 Analytical Operation

- Calibrate the instrument, using a calibration blank and a standard. Refer to Appendix I for the applied levels of concentration.

- All masses which could affect data quality are monitored to determine potential effects from matrix components on the analytes of interest.

- After the calibration has been established, an ICV solution is analyzed to verify the validity of the curve. Measurements for the analytes of interest must be at ±10% of the true value. A re-calibration and re-analysis is required for any analyte which falls outside the control limit.

- Analyze the interference check samples (ICSA and ICSAB) prior to and after the analysis of samples

- Analyze a CCV and a CCB once every 10 analytical samples.

- Dilute samples that exceed the established linear range of the instrument.

Appendix I

Standard Solutions

Elements	Mass	STD, µg/L
Be	9	50
Na	23	500
Mg	26	500
Al	27	400
K	39	500
Ca	44	500
V	51	100
Cr	52	100
Mn	55	150
Fe	54,57	200
Co	59	100
Ni	60	400
Cu	63,65	50
Zn	66	200
As	75	100
Se	82	200
Ag	107	100
Cd	111	50
Sb	121	60
Ba	137	400
Tl	205	100
Pb	208	50
Li *	7	200
B	11	200
Mo	98	200
Sn	118,120	200
U	238	250

The STD is made by diluting 10.0 mL of the ICP/MS stock standard to a 100 mL final volume.

Sr * 88 200

Th * 232 200

HSP 4/20/00

* When requested for specific projects

The ICP/MS stock standard which consists of:

100 µL	ICAL-1
1000 µL	ICAL-2
200 µL	ICAL-3
1000 µL	ICAL-4
100 µL	ICAL-5
250 µL	1000 ppm U
200 µL	1000 ppm B
200 µL	1000 ppm Mo
200 µL	1000 ppm Li
200 µL	1000 ppm Sn
40 µL	1000 ppm Be
150 µL	1000 ppm Se

Note: 50 µL of 20 ppm Sc45, 10 ppm In115 and 10 ppm Bi209 is added to a 10 mL aliquot of standard.

200 uL 1000 ppm ~~Ag~~ Sn*
200 uL 1000 ppm Th*

* When requested for specific projects

NSP
4/20/00

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Appendix II

ICSA and ICSAB Solutions

Element	ICSA (PPM)	ICSAB (PPM)
Aluminum	4	4
Arsenic		0.010
Cadmium		0.010
Calcium	12	12
Chromium		0.020
Cobalt		0.020
Copper		0.020
Iron	10	10
Magnesium	4	4
Manganese		0.020
Molybdenum		0.080
Nickel		0.020
Potassium	4	4
Selenium		0.010
Silver		0.020
Sodium	10	10
Vanadium		0.020
Zinc		0.010

Appendix III

QC Summary Table

Laucks Testing Laboratories

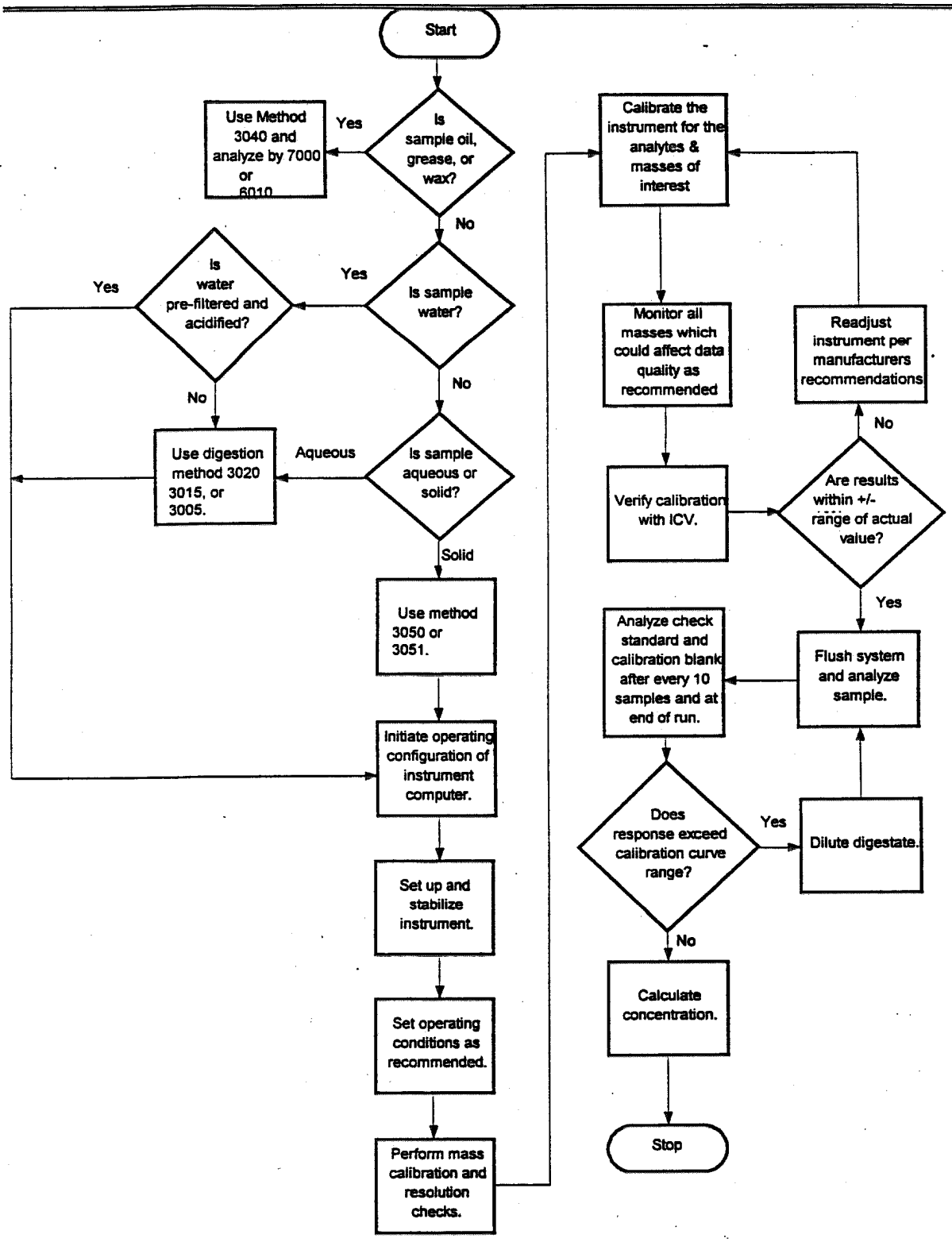
Method SW 846 6020 QA Requirements and Corrective Actions

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Mass Calibration Check	must be performed in mass regions of interest and be within ±0.1 amu of the actual value.	Mg 23.90-24.10 Rh 102.80-103.00 Pb 207.90-208.10	Beginning of each analysis.	Perform new mass calibration.	Instrument Logbook
Resolution Check	< 0.9 amu full width at 10% peak height	< 0.9 amu full width at 10% peak height	Beginning of each analysis.	Adjust resolution.	Instrument Logbook
Initial Calibration	Blank and at least one standard	Blank and one standard	Beginning of each analysis	NA	In the raw data and/or on FORM 14.
Initial Calibration Verification	±10% of true value. Made from an independant source.	±10% of true value. Made from an independant source..	Immediately following calibration.	Recalibrate and reverify.	Form 2, in the raw data
Initial Calibration Blank	Values must be < 3x the IDL for each element.	Values must be < CRDL	Immediately following ICV.	Recalibrate, reverify, and rerun the ICB.	Form3, in the raw data
Continuing Calibration Verification	±10% true value.	±10% true value. Analyte levels are at the mid-range of the calibration.	Every 10 samples and end of run.	Recalibrate and rerun affected samples.	Form 2, in the raw data
Continuing Calibration Blank	Values must be < 3 times the IDL for each element.	Values must be < CRDL	Immediately following CCV.	Recalibrate and rerun affected samples.	Form 3, in the raw data
Method Blank	< CRDL or <5% of regulatory limit or any sample	< CRDL or <5% of regulatory limit or any sample	One/batch	Redigest samples	Form 3, in the raw data.

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Internal Standards	1.Samples: 30 % - 120 % of the initial calibration standard 2. Instrument Check Standards: 80 -120 of the initial calibration standard.	1. Samples 30 % - 120 % of the initial calibration blank. 2. Instrument Check Standards: 80 -120 of the initial calibration blank.	1. All samples 2. All instrument check standards	1. dilute and reanalyze. 2. recalibrate, re-analyze the affected samples.	In the raw data.
Serial Dilution	within ±10% of the original value if the analyte conc. is > 100 x the IDL.	within ±10% of the original value if the analyte conc. is > 100 x the IDL.	One/batch	Flag data with an "E".	Form 9,.in the raw data
Duplicate, % Differenc	± 20% for analyte values greater than 100 times the IDL.	± 20% or current QC database criteria.	5% or per batch	reanalyze digestates, if still fail, consult QCO	Form 6, in raw data, or database report
Matrix Spike Recovery		75-125% or current QC database criteria.	5% or per batch	Consult QCO	Form 5A, in raw data, or database report
Post-Digestion Spike	75%-125%	75%-125%	5% or per batch	dilute and re-analyze or MSA	Form 5B, in the raw data
Laboratory Control Sample	One/batch, no acceptance criteria	LCSW: 80%-120% LCSS: Manufacturer Specs.	One/batch	Redigest samples.	Form 7, in raw data, or database report
ICSA and ICSAB	±20% true value of analytes, or ± the CRDL.	±20% true value of analytes, or ± the CRDL.	Beginning and end of run	reanalyze affected samples	Form 4, or in raw data.

Appendix IV

Flow Chart


Start
Is sample oil, grease, or wax?
Yes
Use Method 3040 and analyze by 7000 or 6010
No
Is sample water?
Yes
Is water pre-filtered and acidified?
Yes
No
Use digestion method 3020 3015, or 3005.
No
Is sample aqueous or solid?
Aqueous
Solid
Use method 3050 or 3051.
Initiate operating configuration of instrument computer.
Set up and stabilize instrument.
Set operating conditions as recommended.
Perform mass calibration and resolution checks.
Calibrate the instrument for the analytes & masses of interest
Monitor all masses which could affect data quality as recommended
Verify calibration with ICV.
Are results within +/- range of actual value?
No
Readjust instrument per manufacturers recommendations
Yes
Flush system and analyze sample.
Analyze check standard and calibration blank after every 10 samples and at end of run.
Does response exceed calibration curve range?
Yes
Dilute digestate.
No
Calculate concentration.
Stop

Appendix V

Routine Reporting Limits

SW 846 6020 Reporting Limits

Element	Reporting Limit, µg/L	Element	Reporting Limit, µg/L
Be	0.5	Cr	1.
Co	0.5	Ni	0.5
Cu	0.5	Zn	5.
As	1.	Se	1.
Ag	0.5	Cd	0.5
Mn	1.	Sb	1.
Ba	0.5	Tl	0.5
Pb	0.5	Zn	1.0

Reporting limits are approximately 2-10 times the instrumental MDL. The MDL is based on samples prepared using SW 846 3015. Values actually reported may be less than the routine reporting limits but above our method detection limit.

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-7501

Title: **Operation of the PE FIMS 400 for the Analysis of Hg**

Revision history:

Number	Date
1	02/04/98
0	02/14/96

UNCONTROLLED

Revised by: Bill Lundberg
Bill Lundberg, Inorganics Division Manager
Date: 2/5/98

Approved by: Harry Romberg
Harry Romberg, QC Officer
Date: 2-5-98

Approved by: Karen J. Kotz
Karen Kotz, Laboratory Director
Date: 2/5/98

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1. Introduction and Scope

1.1 Method Description:

1.1.1 This SOP covers the cold vapor analysis of Hg by the Perkin-Elmer Flow Injection Mercury System, Model 400 (FIMS 400). Sample handling and preparation are as required by SW 846 methods 7470 and 7471. This SOP is also valid for the preparation and analysis of samples under CLP protocol when used in conjunction with the SOW ILM04.0. Where the requirements of these procedures differ, the most stringent requirements are used. The requirement of analyzing soil samples in triplicate under 7471 is not used except on request.

1.1.2 After preparation and digestion to convert Hg to its ionic form, 0.5 mL of sample is introduced into a stream of carrier solution (3% HCl), mixed with a $SnCl_2$ suspension to reduce the Hg ion to its metallic form, which is then carried into an adsorption tube through which passes light at a wavelength of 253.7 nm. The adsorption is then measured.

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

1.2 Sample Collection, Sample Storage, Holding Times

1.2.1 Samples are stored either in glass or plastic. The holding time is 28 days from collection. Soil samples are stored at 4° C±2° C. Water samples are stored at room temperature. Water samples are preserved by the addition of HNO_3 to a pH <2.

1.3 Definition of Terms

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

1.3.2 LFB - Laboratory Fortified Blank - A sample of DIW to which a known amount of Hg is added.

1.3.3 CCB - Continuing Calibration Blank - This is the same acronym used in the CLP program. This is a blank which is analyzed immediately after the CCV (almost always

after every 10 samples and at the end of the analytical run) during the analysis sequence to determine whether the instrument or system has maintained a stable baseline.

1.3.4 CCV - Continuing calibration verification. - This is the same acronym used in the CLP program. This is a standard analyzed at some prescribed frequency (almost always after every 10 samples and at the end of the analytical run) during the analysis sequence to determine whether the instrument or system has remained in calibration.

1.3.5 CLP - Contract Laboratory Program - The USEPA program that contracts with laboratories to provide laboratory services. The term has come to mean a much broader set of methods and deliverables. In context of this SOP, CLP means procedures or operations which are detailed in the CLP contract and which are extended to a broader working definition.

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

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

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

1.3.9 ICV - Initial calibration verification - is a standard which is analyzed at the start of each analytical run that is compared to the initial multi-point calibration to determine whether the instrument calibration is accurate. This verification standard is from a source different from that used to make the calibration standards

1.3.10 IDL - Instrument detection limit. The lowest concentration of a target analyte that will yield a signal:noise ratio of least 3x. Determined quarterly using blanks to which a known quantity of standard has been added. The IDL is determined by analyzing 7 replicates spiked at 2-5 times the expected IDL on three non-consecutive days. The sum of the standard deviations of the three runs is the IDL.

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

1.3.12 MDL standard - Method detection limit standard - A standard prepared so that the concentration of Hg is 1-5 times the anticipated MDL

1.3.13 LCS - Laboratory Control Sample - A sample containing a known quantity of Hg used to verify digestion and analysis. LCSW indicates a water sample. It is used interchangeably with ICV. LCSS indicates a soil sample.

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

1.3.15 PBW - A blank carried through the sample preparation and digest procedures.

2. Equipment, Standards and Reagents

2.1 Equipment

2.1.1 Equivalent equipment may be used where appropriate.

FIMS 400 automatic Hg analyzer.
Perkin Elmer AS-90 autosampler.
Epson LQ300 dot-matrix printer.
Wells 18"X24" hot water bath.
BOD bottles
Analytical balance
Micropipettes: 25, 50, 100, 200, 500 and 1000 µl.
50 mL and 15 mL centrifuge tubes - polystyrene.

2.2 Standards

2.2.1 All standards must be prepared in 0.15% HNO_3.

2.2.2 Intermediate Hg solution: Dilute 100 µl of 1000 ppm Hg standard solution to 100 mL in DIW to which 0.15 mL HNO_3 has been added. The identification number of the standard solution used and the dilution must be entered on the digestion log (Appendix II). This solution must be prepared daily. Since the intermediate and working standards are prepared daily, they are not recorded in the standards logbook.

2.2.3 0.2 µg/L standard - Place 100 mL DIW in a BOD bottle. Add 20 µl stock Hg solution

2.2.4 0.5 µg/L standard - Place 100 mL DIW in a BOD bottle. Add 50 µl stock Hg solution.

2.2.5 1.0 µg/L standard - Place 100 mL DIW in a BOD bottle. Add 100µl stock Hg solution.

2.2.6 2.0 µg/L standard Place 100 mL DIW in a BOD bottle. Add 200 µl stock Hg solution.

2.2.7 5.0 µg/L standard - Place 100 mL DIW in a BOD bottle. Add 500 µl stock Hg solution.

2.2.8 10.0 µg/L standard - Place 100 mL DIW in a BOD bottle. Add 1000 µl stock Hg solution.

2.2.9 ICV solution - Add 100 mls DIW to a BOD bottle. Add sufficient stock ICV solution to produce a concentration of 1.0 to 5.0 µg/L.

2.2.10 CCV/CCB/LFB - In setting up the run, use the 5.0 µg/L standard for the CCV and LFB; for the CCB use the calibration blank.

2.2.11 MS/MSD - Add 100 µl intermediate Hg solution to the samples being spiked. When performing Hg under CLP protocols use 100 µl intermediate Hg solution and perform MS/Duplicate.

2.2.12 LCSS - Laboratory control soil sample. Concentrations and limits are certified by the manufacturer.

2.3 Reagents:

2.3.1 DIW - deionized water free of impurities.

2.3.2 H_2SO_4 (sulfuric acid) - concentrated, reagent grade

2.3.3 HNO_3 (nitric acid) - concentrated, reagent grade

2.3.4 HCl (hydrochloric acid) - concentrated, reagent grade

2.3.5 Stannous chloride - Add 11 g. $SnCl_2 \cdot 2H_2O$ (stannous chloride dihydrate) to 1 L DIW to which has been added 30 mL concentrated HCl.

2.3.6 $KMnO_4$ (potassium permanganate) - Dissolve 100 g. $KMnO_4$, marked "suitable for Hg determination", in 2 L DIW. This is a saturated solution. Sufficient undissolved $KMnO_4$ should be allowed remaining in the bottom of the bottle to assure a saturated solution.

2.3.7 $K_2S_2O_8$ (potassium persulfate) - Dissolve 50 g. $K_2S_2O_8$, marked "suitable for Hg determination", in 1 L DIW. This is a saturated solution. Sufficient undissolved $K_2S_2O_8$ should be allowed remaining in the bottom of the bottle to assure a saturated solution.

2.3.8 $NaCl/NH_2OH{\cdot}HCl$ (sodium chloride/hydroxylamine hydrochloride) - Dissolve 240 g NaCl (table salt is generally used) and 240 g. $NaCl/NH_2OH{\cdot}HCl$ (reagent grade marked "suitable for Hg determination") in 2 L DIW. Contamination has been experienced with this solution. For this reason a 50% solution should be analyzed by the FIMS 400 whenever a new bottle is made up. If the absorbance (peak height) exceeds .004 the container of $NaCl/NH_2OH{\cdot}HCl$ should be marked "Contaminated" and disposed of.

2.3.9 Carrier solution - The carrier is a 3% HCl solution.

3. Safety Precautions and Waste Disposal:

3.1 Safety Precautions

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

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

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

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

3.1.5 Because of the toxic nature of Hg vapor, inhalation or skin contact should be avoided.

3.1.6 The usual precautions should be taken in handling acids.

3.1.7 $SnCl_2$ is a skin and eye irritant; avoid contact.

3.1.8 The addition of hydroxylamine after digestion releases gas, including chlorine. Avoid inhalation; use hood if necessary.

4. Calibration and Quality Control

4.1 Method Detection Limit Study (MDL).

4.1.1 MDL studies for Hg water samples and soil samples are performed annually. Consult the current SOP on MDL determinations for specifics.

4.2 Initial Multi-Point Calibration:

4.2.1 Concentrations:

Standard Name	Concentration
S0	0.0 µg/L
S0.2	0.2 µg/L
S0.5	0.5 µg/L
S1.0	1.0 µg/L
S2.0	2.0 µg/L
S5.0	5.0 µg/L
S10.	10.0 µg/L

Standards are made fresh daily from the intermediate standard in 0.15% HNO_3.

4.2.2 The calibration blank, the ICV, the LCS, the LFB, the ICB and all standards are digested along with the samples being analyzed.

4.3 Calibration Criteria

4.3.1 Calibration curve - CC must be greater than 0.995.

4.3.2 ICV - For CLP analyses the limits are 80-120 %, for SW 846 the limits are 90-110%.

4.3.3 ICB - Limitations presently in use are -0.2 µg/L to 0.2 µg/L .

4.3.4 A PBW and an LFB are run immediately following the successful calibration. The limitations on the PBW are the same as the limitation on the ICB. The limitations on the LFB are as set forth in QC-DB.

4.3.5 If the above criteria are not met, recalibration must be performed.

4.4 Continuing quality control:

4.4.1 A CCV followed immediately by a CCB must be run every 10 samples and after the last sample. The ICB, PBW and LFB count as samples for this purpose.

4.4.2 Criteria - For CCV's the limitations are 80% to 120%. For blanks the limitations are -0.2 µg/L to 0.2 µg/L .

4.4.3 Corrective action - If a CCV or CCB are out of control, a recalibration must be performed followed by a reanalysis of all samples since the last previous successful CCV and/or CCB.

4.5 Matrix Spike

4.5.1 A sample is chosen at random from the samples to be analyzed, and 100 µl of intermediate standard is added. The spike level is then 1.0 µg/L. The analyst should attempt to avoid selecting samples which are identified by the client as blanks. As the purpose of the matrix spike is to test the system under "typical" conditions, the analyst may also avoid selecting the most difficult sample of the batch for spiking. It is not always required that a matrix spike analysis be performed with each preparation/analysis batch, however, the minimum frequency for MS analysis is 1 each per 20 samples per matrix. This will be best accomplished by running one with every batch for many analyses. This matrix spike sample is used to evaluate the matrix effect of the sample upon recovery of the analytes. The recovery of spike analytes is calculated as follows:

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

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

4.5.2 The recovery criteria are listed in QC_DB and will change from time to time.

4.5.3 Corrective action

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

4.6 Matrix Spike Duplicate

4.6.1 The compound recovery criteria are identical to those for the matrix spike sample. In addition, the matrix spike duplicate is used measure method precision. This is done by computing the relative percent difference (RPD) between the matrix spike and matrix spike duplicate recovery values. This calculation is as follows:

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

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

4.6.2 RPD control limits are listed in QC_DB and will change from time to time.

4.7 Sample Duplicate

4.7.1 Sample duplicates are required when CLP practices are employed, or when the method specifically calls for duplicates. At least one duplicate sample per 20 samples per matrix is required when matrix spikes are being performed.

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

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

4.7.2 The RPD control limits listed in QC_DB and will change from time to time.

4.7.3 Corrective action

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

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

5. Operation Procedures:

5.1 Digest - BOD bottles - Soil or Water:

5.1.1 Digestion log (Appendix II): The digestion log should show the sample numbers, the date, the name of the analyst, the time placed in the hot water bath, the time when the temperature reached 95± 5° C, and the time removed from the hot water bath. One should be prepared for each run. An example is attached as Appendix II. The numbers of each BOD bottle must be placed in the leftmost column of this form to identify which sample is in each BOD bottle.

5.1.2 Preparation of standards:

5.1.2.1 Prepare an intermediate standard daily by adding 100 µl of the 1000 mg/L stock standard to 100 mL of 0.15% HNO_3 in DIW, giving a concentration of 1.0 mg/L of Hg.

5.1.2.2 Place 100 mL DIW in each of 9 BOD bottles.. Mark these BOD bottles with the concentration of each standard in µg/L , plus one marked "Blank", one marked "ICV" and one more marked ICB.

5.1.2.3 Add from the intermediate standard the following amounts to the following BOD bottles:

0 µg/L	0 µl
0.2 µg/L	20µl
0.5 µg/L	50 µl
1.0 µg/L	100 µl
2.0 µg/L	200 µl
5.0 µg/L	500 µl
10.0 µg/L	1000 µl

5.1.2.4 To the ICV BOD bottle add that amount of the ICV standard which will produce a concentration from 1.0 µg/L to 5.0 µg/L .

5.1.2.5 The standards, the ICV and the ICB must be digested along with the PBW, LFB and samples.

5.2 Digestion - Water Samples

5.2.1 Place 100 mL DIW in the PBW BOD bottle.

5.2.2 Place 100 mL DIW + 100 µl intermediate standard in the LFB BOD bottle.

5.2.3 Place 100 mls sample in the BOD bottle designated for that sample, being sure to place 100 mL sample in the designated duplicate, MS and MSD BOD bottles for the sample involved. TCLP samples and spikes should be diluted 20 mL/100 mL (dilution factor of 5) in DIW. The TCLP spikes should be diluted 2 mL/100 mL in DIW.

5.2.4 Add the required quantity (See Sec. 4.5.1) of intermediate standard to the MS and MSD BOD bottles.

5.2.5 Add the following reagents to each BOD bottle:

5 mL concentrated H_2SO_4
2.5 mL concentrated HNO_3
15 mL $KMnO_4$ solution
8 mL $K_2S_2O_8$ solution

5.2.6 The $KMnO_4$ color must persist for 15 minutes. If it does not, more $KMnO_4$ must be added.

5.2.7 Place all the BOD bottles, being careful to keep them in order, in the hot water bath in a hood, and record the time on the digest log. Monitor the temperature of the hot water bath and record the time when the temperature reaches 90° C. Continue to heat the water bath, keeping the bath at 95 ± 5 C for two hours.

5.2.8 Remove the BOD bottles from the hot water bath and allow to cool.

5.2.9 Add 6 mL $NaCl/NH_2OH{\cdot}HCl$ solution and mix until the mixture clears. If some undissolved permanganate remains, it will usually disappear on standing after a few minutes.

5.3 Digestion - Soil samples:

5.3.1 Place 100 mL DIW in the PBW BOD bottle.

5.3.2 Place approximately 0.5 g., or, for certain CLP and 846 analyses, approximately 0.2 g of sample in each of the BOD bottles to which that sample applies. Record the exact weight on the digestion log.

5.3.3 Add 3.75 mL HCl , 1.25 mL HNO_3 and 5 ml DIW to each BOD bottle.

5.3.4 Place all BOD bottles in a hot water bath in a hood that is at 95° C ± 5° C. Maintain the heat at this level for 2 minutes.

5.3.5 Cool for a few minutes and add 50 mL DIW and 15 mL $KMnO_4$. Mix.

5.3.6 The $KMnO_4$ color must persist for 15 minutes. If it does not, more $KMnO_4$ must be added.

5.3.7 Place all BOD bottles in a hot water bath that is at 95° C ± 5° C. Record the time on the digest log. Maintain the hot water bath at this temperature for 30 minutes. Remove the BOD bottles and allow to cool.

5.3.8 Add 6 mL $HCl/NH_4NH_2OH{\cdot}HCl$ solution and mix to remove color.

5.3.9 Add 50 mL DIW. Allow to cool.

5.4 Analysis on FIMS 400.

5.4.1 This is a programmed analysis. The method name is "hg_cv" (Appendix V). This is a 6 point curve with a blank correction. The development of the calibration curve, including the ICV and ICB, is automatic. If the calibration is unsuccessful, recalibration is attempted. If a standard is seriously out of line, it is ignored by the Perkin-Elmer software. The 5.0 µg/L standard is used for CCV's; the calibration blank for CCB's, both of which are programmed to be run every 10 samples. If a CCV or a CCB fails to meet quality control specifications, the system recalibrates and reruns all samples since the last preceding valid CCV or CCB. A CCV and a CCB are also run after the last sample in the run.

5.4.2 Sample Information sheet - Appendix IV.

5.4.2.1 Click the automatic analysis button on the screen which appears after activating the program.

5.4.2.2 Click the "Sample Information" button on the toolbar.

5.4.2.3 A blank sample information sheet appears. Previous sample information sheets may be obtained by activating the File - Open menu, designating "Sample Information" from the dialog which appears. The sample information sheet should be filled in to tell the computer which auto-sampler locations to go to.

5.4.2.4 Sample ID column - Some typing can be saved by double clicking on the column title. On the resulting form the job order may be typed in and the sample numbers will then be incremented by 1 for the autosampler locations indicated. The sample ID's will then be filled in. Sample: "9802094-0001..0002. .".

5.4.2.5 Sample weight column - For soil samples the weight of the samples should be filled in. At the top, the volume units should be filled in "mL", the weight units "g".

5.4.2.6 Sample units column - Typing may be saved by double clicking on the column title and indicating the units on the dialog box which appears, and indicating the AS locations involved. In the case of water samples this will ordinarily by "µg/L ". For soil samples the button "weight:weight" should be clicked and the desired units specified in the dialog box selected. If the dialog box has been properly filled in, the machine is programmed to compute the proper units (e.g. mg/kg). It should be kept in mind that this is on an as received basis, not a dry basis.

5.4.2.7 User dilution column - Where a dilution is involved (e.g. TCLP analyses) the dilution factor should be entered. As in the sample weight and sample units columns, above, typing may be saved by double clicking the column title.

5.4.2.8 The autosampler tray being used at present is "Tray B".

Sampler Location	Size	Contents
1	50 mL tube	Calibration Blank
2	50 mL tube	0.5 µg/L standard
3	50 mL tube	1.0 µg/L standard
4	50 mL tube	2.0 µg/L standard
5	50 mL tube	5.0 µg/L standard
6	50 mL tube	10.0 µg/L standard
7	50 mL tube	ICV
8	50 mL tube	ICB
9	15 mL tube	PBW or LCSS
10	15 mL tube	LFB or sample
11-109	15 mL tubes	Samples

5.4.3 All tubes should be marked with their contents before being filled. Then the tubes are filled from the BOD bottles and placed in the autosampler locations designated either by the method or by the sample information sheet.

5.4.4 At the beginning of each day of analysis, the following steps are taken to prepare the instrument:

5.4.4.1 The flows of the carrier solution and the reductant solution (the stannous chloride solution) are measured by the methodology suggested in the Perkin-Elmer handbook "Setting Up and Performing Analyses", 2.4.2-2.4.4. The rinse container (autosampler location 0) is rinsed and filled with 3% HCl solution. This is then analyzed using the location button in the analysis window of the automated analysis window, and the curve produced displayed through the curve button in the toolbar. The curve should be flat and less than 0.0001 absorbance units.

5.4.5 Analysis - The analysis is ordinarily started by clicking the "Analyze All" button in the Automated Analysis window. If only calibration is desired, the "Calibrate" button may be used. If samples are to be analyzed by an already existing calibration curve use the "Analyze Samples" button.

5.4.5.1 Once the analysis has been started the autosampler places the sampling probe in the sample/calibration cup. The peristaltic pump begins pulling sample through the sample loop. After the line has been purged, the injection valve cycles, and injects the sample into the carrier flow. This flow is then combined

with the SnCl2 and mixed. Next it goes to a liquid/vapor separator. The liquid goes to waste and the vapor is transported into the adsorption cell of the spectrophotometer. All samples and standards are measured in duplicate.

5.4.6 Run log - A run log will be printed as the analysis progresses. The print program is designed to transmit the data to be printed to the printer one page at a time. Thus several autosampler locations will be analyzed before anything appears on the printer. If anything else is printed (e.g. the sample information sheet) while the automated analysis window is open, the computer must be rebooted, or a run log will not be printed.

5.4.7 Additional samples may be analyzed after the run designated on the sample information sheet by using the "Select Location" button on the "Analyze" page of the Automated Analysis window. The autosampler locations should be filled in the resulting dialog.. The instrument will then analyze the samples indicated. This may be used to reanalyze the samples with questionable results, or to analyze diluted samples.

5.4.8 Dilution - Where analysis results are above the high standard (10.0 µg/L) an aliquot of the sample diluted with the calibration blank may be analyzed as set forth in 5.4.7 above.

6. Reports

6.1 Data Packet Organization

6.1.1 A report is prepared using the reformat procedure on the main menu. The reformatted design is entitled "Summary". It is transferred to a floppy disk, which is then imported to a spreadsheet using Microsoft Excel 5.0. This must then be formatted into the form included in Appendix III.

6.1.2 The final data packet is to include the following:

1) The report (See Appendix III).
2) The QC_DB Report Form
2) The digestion log (Appendix II).
4) The run log, if required.

6.1.3 All results for quality control tests are entered into QC_DB. A summary report of all data entered must be included in the data packet. The routine minimum is a method blank report, an MS/MSD or MS/duplicate report and a SRM/LSCW.

6.2 Data Qualifying Flags

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

6.3 Control charts

6.3.1 The recovery values for the LCS/SRM are plotted on control charts.

Appendix I - QC Summary Table

Laucks Testing Laboratories
Method 245.1,7471,7470 QA Requirements and Corrective Actions

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Initial Calibration	CC > 0.995	CC > 0.995	once per batch	recalibrate	run log
Initial Calibration Verification	90-110 % SW846 80-120% 245.1	Per the method	one per 20	recalibrate	run log, report
Initial Calibration Blank	Below R.L.	Below R.L.	one per sequence	recalibrate	run log, report
Continuing Calibration Verification	80-120 %	80-120 %	At the beginning,every 10 samples and after the last sample	recalibrate and rerun samples since last CCV	run log
Continuing Calibration Blank	Below R.L.	Below R.L.	At the beginning,every 10 samples and after the last sample	recalibrate and rerun samples since last CCB	run log
Matrix Spike Recovery	SW-846 See QC_DB. CLP 75-125%	See QC_DB	Every 20 samples	Consult QC officer. Flag data or reanalyze.	QC_DB
MS/MSD RPD	See QC_DB	See QC_DB	Every 20 samples	Consult QC officer. Flag data or reanalyze.	QC_DB
Duplicate % Difference	20% RPD CLP	See QC_DB	Every 20 samples	Consult QC officer. Flag data or reanalyze.	QC_DB

Laboratory Fortified Blank (LFB)	85-115 %	See QC_DB	Once per 20	Recalibrate	Run log
Standard Reference Material (SRM) Recovery	Soil Manufacture's limits Water See ICV	Manufacturer's limits	Once per 20	Recalibrate	Run log

Appendix - II - Sample Digestion Log

Date		Bottles into water bath (time)	
Analyst		Water bath at 95°C (time)	
Standard ID	ME-	Bottles out of water bath (time)	
ICV No	ME-	Spike = 100 µl of Std	

No.	Sample ID	Sample size g , mL	Final volume,mL	Comments	Bottle #
	S0 ppb		100 ml		
	S0.2 ppb 20 µl of Std		100 ml		
	S0.5 ppb 50 µl of Std		100 ml		
	S1.0 ppb 100 µl of Std		100 ml		
	S2.0 ppb 200 µl of Std		100 ml		
	S5.0 ppb 500 µl of Std		100 ml		
	S10.0 ppb 1000 µl of Std		100 ml		
	ICV		100 ml		
	ICB		100 ml		
	LCSW		100 ml		
	CRA		100 ml		
	PBW		100 ml		
			100 ml		
			100 ml		
			100 ml		
			100 ml		
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			100 ml		
			100 ml		
			100 ml		
			100 ml		
			100 ml		

$NH_2OH \cdot HCl$	NaCl	$K_2S_2O_8$	$SnCl_2 \cdot 2H_2O$	$KMnO_4$
REA1-6-3	REA1-7-9	REA1-5-7	REA1-5-3	REA1-5-4

Appendix III - Example Report Form

Hg in Water and TCLP
Digested 1/7/98
Analyzed 1/8/98

Sample	Time	Value		% Rcvry	RPD	Flag	Comment
Calib Blank	11:13:29		µg/L				
S0.2	11:16:23		µg/L				
S0.5	11:19:19		µg/L				
S1.0	11:22:19		µg/L				
S2.5	11:25:17		µg/L				
S5.0	11:28:15		µg/L				
S10.0	11:31:16		µg/L				
ICV1 ME7-23-2=4	11:34:21	4.028	µg/L	101%			
ICB	11:37:21	0.001	µg/L				
CCV1=5	11:40:16	5.015	µg/L	100%			
CCB1	11:43:10	0.007	µg/L				
LCSW1	11:46:04	4.067	µg/L	102%			
CRA	11:48:57	0.226	µg/L				
PBW	11:51:55	0.003	µg/L				
12611-11	11:54:50	2.215	µg/L				did not report
12679-02	11:57:47	0.007	µg/L				
12679-02dup	12:00:44	0.017	µg/L				
12679-02ms Spk=1	12:03:42	12.842	µg/L	1284%		N	see below
12679-03	12:06:42	0.083	µg/L				
12679-04	12:09:34	-0.016	µg/L				
CCV2=5	12:12:28	5.138	µg/L	103%			
CCB2	12:15:24	0.013	µg/L				
12679-05	12:18:16	0.026	µg/L				
12679-06	12:21:11	0.029	µg/L				
12679-07	12:24:04	0.002	µg/L				
12679-08	12:26:58	8.239	µg/L				see below
12679-09	12:29:52	23.125	µg/L				did not report
12679-10	12:32:46	0.209	µg/L				did not report
12725-01	12:35:44	0.367	µg/L				
12725-02	12:38:39	-0.226	µg/L				
12725-02dup	12:41:36	-0.419	µg/L				
CCV3=5	12:44:32	4.945	µg/L	99%			
CCB3	12:47:26	0.014	µg/L				
12725-02ms Spk=200	12:50:25	199.903	µg/L	100%			
12184-5	12:53:22	1.436	µg/L				
12184-5dup	12:56:18	1.433	µg/L				
CCV4=5	12:59:16	5.111	µg/L	102%			
CCB4	13:02:10	-0.028	µg/L				
12679-02ms (rerun)	13:08:05	0.959	µg/L	96%			
12679-02ms (10x dil)	13:10:59	-0.646	µg/L				did not report
12679-08 (rerun)	13:13:49	0.001	µg/L				
12679-09 (10x dil)	13:16:40	0.211	µg/L				did not report
CCV5=5	13:19:35	4.940	µg/L	99%			
CCB5	13:22:32	0.011	µg/L				

Appendix IV - Example Sample Information Sheet

Autosampler Loading List

Sample Information File: 020598S.SIF
Methods: Soils

Location	Elements	Solution
0	Hg	Wash Solution
1	Hg	Calib Blank
	Hg	CCB1: 0.0000 µg/L
	Hg	CCB: 0.0000 µg/L
2	Hg	S0.2: 0.2 µg/L
3	Hg	S0.5: 0.5 µg/L
4	Hg	S1.0: 1.0 µg/L
5	Hg	S2.0: 2.0 µg/L
6	Hg	S5.0: 5.0 µg/L
	Hg	CCV1: 5.0000 µg/L
	Hg	CCV: 5.0000 µg/L
7	Hg	S10.0: 10.0 µg/L
8	Hg	ICV: 4.0000 µg/L
9	Hg	ICB: 0.0000 µg/L
10	Hg	Sample: CRA
11	Hg	Sample: PBW
12	Hg	Sample: LCSS
13	Hg	Sample: 9801614-01
14	Hg	Sample: 614-01ms
15	Hg	Sample: 614-01msd
16	Hg	Sample: 549-02 (10x)
17	Hg	Sample: 549-03 (10x)
18	Hg	Sample: 549-03(20x)

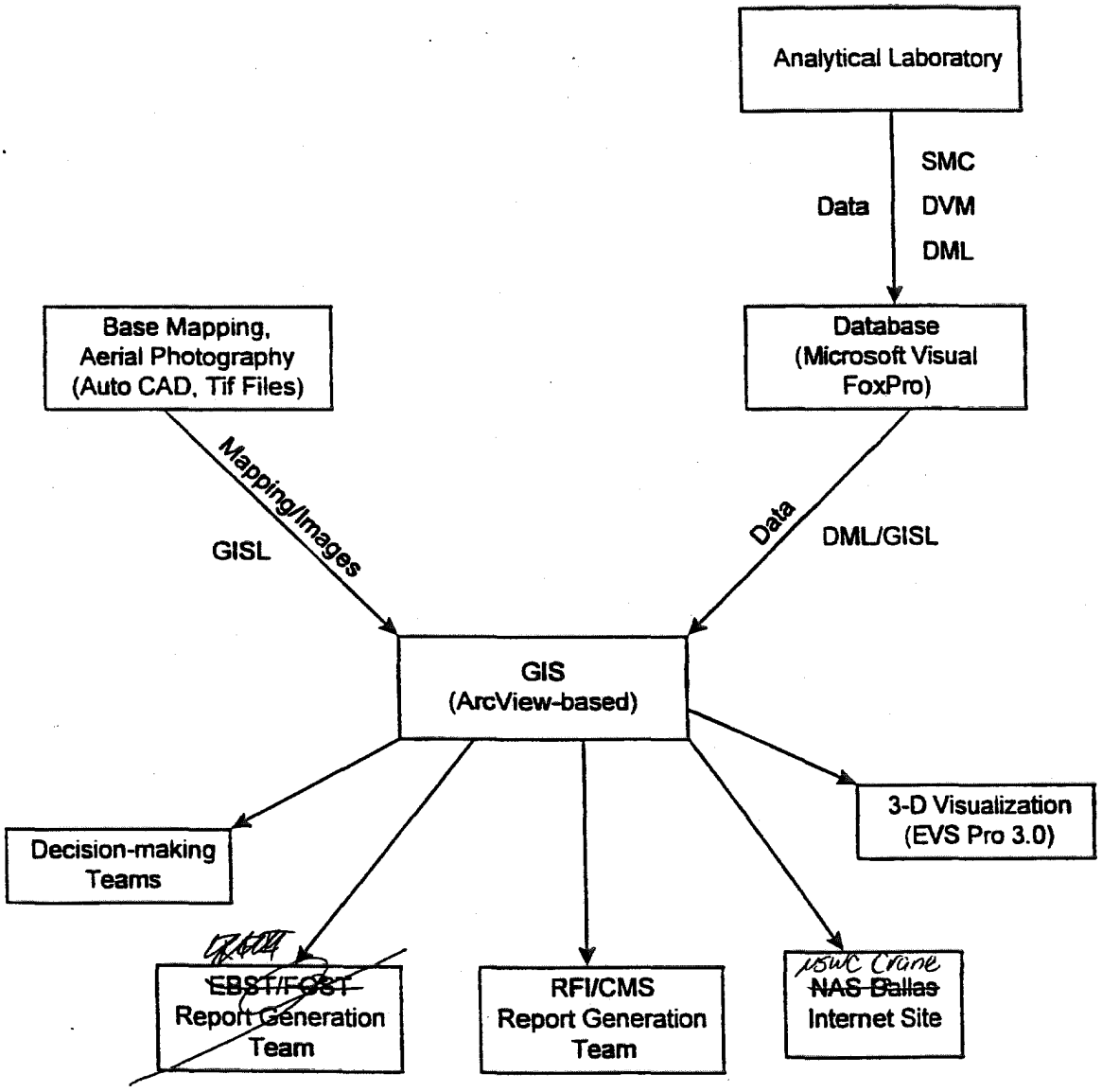
ATTACHMENT 1

DATA CATALOG

ATTACHMENT 2

THE DATA FLOW PROCESS

THE DATA FLOW PROCESS



Notes:

SMC = Sample Management Coordinator
DVM = Data Validation Manager
DML = Data Management Leader
GISL - GIS Leader

ATTACHMENT 3

DATABASE CHECKLIST

DATABASE PLANNING CHECKLIST

PROJECT NAME _____ PROJECT NUMBER _____

PROJECT MANAGER _____ PLANNING DATE _____

1 Provide a general description of the project (regulatory authority, media to be samples, approximate number of samples by media, analyses by media, data evaluation tasks required):

_____
_____
_____
_____
_____

2. Provide a general description of the sample nomenclature that will be used for samples collected by Brown & Root Environmental:

_____
_____
_____
_____

3. Will historical data be entered in the database? Yes No

4. Will historical data be used to define the nature and extent of contamination? Yes No

5. Will historical data be used for risk assessment purposes? Yes No

6. How much historical data exists (i.e., number of samples by matrix, analysis by matrix)?

_____
_____
_____
_____

7. In what format will the historical data be provided? Hardcopy Electronic

8. If historical data are in electronic form, what software was used and what is the format?

_____
_____
_____
_____

9. If historical data are in hardcopy form, will Form I's, summary tables, or reports be provided? Copies of historical data will be necessary to generate a budget estimate.

_____
_____
_____
_____

10. Will Quality Assurance review of historical data be necessary? Yes No

11. If Quality Assurance review of historical data is necessary, describe the scope of the Quality Assurance review:

_____
_____
_____
_____

12. Will a GIS database be necessary for the project? Yes No

13. What nomenclature has been (will be) used to identify field duplicate samples?

_____
_____
_____
_____

14 Will field duplicate results be averaged and presented as one result in the data base? Will they be presented as distinct results, or will both the average and the distinct results be presented?

_____
_____
_____
_____

15. How will the average value for duplicate samples be determined on a matrix-specific basis?

_____
_____
_____
_____

16.	Are any unvalidated data to be included in the database?	Yes No
17.	Will unvalidated data be used for defining the nature and extent of contamination?	Yes No
18.	Will unvalidated data be used for risk assessment purposes?	Yes No
19.	Are any field screening data to be included in the database?	Yes No
20.	Will field screening data be used for defining the nature and extent of contamination?	Yes No
21.	Will field screening data be used for risk assessment purposes?	Yes No
22.	Will statistical correlation of laboratory and field screening data be necessary?	Yes No
23.	If a correlation exists between field screening and laboratory data, will the results of regression analysis be used to define nature and extent?	Yes No
24.	If a correlation exists between field screening and laboratory data, will the results of regression analysis be used to support the risk assessment?	Yes No
25.	Will field parameters be included in the database (e.g., pH, conductance, temperature)?	Yes No
26.	Will statistical correlations be necessary for TCLP versus RAS/SAS data?	Yes No
27.	Will statistical correlations be necessary for filtered versus unfiltered samples?	Yes No
28.	Will any other statistical correlations be necessary?	Yes No
29.	Are there wells that have been screened in different aquifers?	Yes No
30.	Will data for various aquifers be segregated by depth?	Yes No
31.	Can the sample nomenclature system be used to identify wells in different aquifers?	Yes No
32.	Will samples from other matrices (soil, sediment, or surface water) be segregated by depth?	Yes No
33.	Can the sample nomenclature system be used to identify depth-specificity?	Yes No
34.	Have any removal actions be performed at the site?	Yes No

If removal actions have been performed, plan and cross-sectional views reflecting the extent of the removal action must be provided.

35. Will any composite sample results be included in the database? Yes No

36. If composite samples are included how will they be used for the nature and extent of contamination?

_____
_____
_____

37. If composite samples are included how will they be used for the risk assessment?

_____
_____
_____

38. Will the site be segregated into Areas of Concern, Solid Waste Management Units, etc? Yes No

39. Is the sample nomenclature adequate for such segregation? Yes No

If the sample nomenclature is inadequate for assigning samples to an AOC or SWMU, the Project Manager or designee must provide a base map of tabular summary clearly delineating the relationship between each sample and each AOC/SWMU.

40. Were any temporal samples collected (e.g., quarterly sampling of wells)? Yes No

41. If temporal samples were collected, how will they be used to define the nature and extent of contamination?

_____
_____
_____

42. If temporal samples were collected, how will they be used to support the risk assessment?

_____
_____
_____

43. Are State, Federal, or Regional criteria to be included in data summary tables? Yes No

44. Identify the criteria that must be presented in the summary tables.

_____
_____
_____

45. Will State, Federal, or Regional criteria be used to select COPCs? Yes No

46. Identify the criteria to be used as COPC selection tools.

_____
_____
_____

47. Are filtered and unfiltered surface water samples differentiated? Yes No

48. If such samples are differentiated, how?

_____
_____
_____

49. Which of these samples will be used for the human health risk assessment?

Surface Water Filtered Unfiltered
Groundwater Filtered Unfiltered

50. Which of these samples will be used for the ecological assessment?

Surface Water	Filtered	Unfiltered
Groundwater	Filtered	Unfiltered

51. Will background data be included in the database? Yes No

52. How are background samples identified?

_____
_____
_____

53. Will background results be used to support selection of COPCs? Yes No

53. What statistical analyses will be required for the background data?

_____
_____
_____

54. Will background data be segregated by depth? Yes No

55. What background matrices must be segregated by depth?

_____
_____
_____

56. What format will be used for data presentation (e.g., appendices and summary tables, comprehensive text tables, tag maps, isoconcentration contours, etc.)?

_____
_____
_____

ATTACHMENT 4

SAMPLE TRACKING REQUEST FORM

Sample Tracking and Data Management at Project Inception

PROJECT START-UP CHECKLIST

ATTACHED IS A PROJECT START-UP CHECKLIST (CAN BE FOUND IN DATA MANAGEMENT ROOM). WHENEVER A NEW PROJECT IS STARTED THE TOP PART SHOULD BE FILLED IN. A COPY SHOULD BE RETURNED TO THE DATA MANAGEMENT ROOM. KEEP ORIGINAL FOR YOUR RECORDS TO KEEP TRACK OF WHAT HAS BEEN PROVIDED. IMSG WILL CHECK OFF WHEN ALL INFORMATION IS RECEIVED

FOLLOWING THIS PROCESS WILL IMPROVE THE FOLLOWING:

- TURN-AROUND TIME FOR DELIVERABLES NEEDED WHEN ALL RESULTS HAVE BEEN RECEIVED.
- CONFIDENCE THAT ALL SAMPLE RESULTS HAVE BEEN RECEIVED
- CONSISTENCY OF SAMPLE NOMENCLATURE
- CORRECTNESS OF SAMPLE ATTRIBUTES
- REVIEW OF INVOICES
- ENABLE IMSG PERSONNEL TO BETTER TRACK UPCOMING WORKLOAD

PROJECT START-UP CHECKLIST
INFORMATION NEEDED TO CREATE NEW DATABASE

PROJECT NAME:_____

CTO #:_____ JOB #:_____

PROJECT MANAGER/CONTACT:_____

LABELS: Y / N DUE DATE:_____

VALIDATE: Y / N / L DUE DATE:_____

COMBINE WITH HISTORICAL DATA: Y/N

SAMPLE DATA CHECKLIST:

_____ SAMPLE NUMBERS AND ANALYSES (LOCATIONS,DEPTHS)
_____ SECTION OF WORKPLAN PERTAINING TO SAMPLE NOMENCLATURE
_____ LABORATORY/BOTTLE REQUIREMENTS
_____ LAB SPECS
_____ COC'S
_____ SAMPLE LOG SHEETS
_____ DUPLICATE ID'S / ORIGINALS
_____ SURVEY DATA / SAMPLE LOCATION MAPS
_____ BREAKDOWN OF PROJECT BY SITE / MATRIX FOR FUTURE PRINTOUTS
_____ TABLE HEADERS (SEE EXAMPLE)

TO BE COMPLETED BY IMSG:

_____ FINAL RESULTS GIVEN TO _____ (PM/IMSG)
DATE:_____
_____ SAMPLE DATA LOADED INTO NEW/EXISTING PROJECT DATABASE
_____ RESULTS LOADED INTO NEW/EXISTING PROJECT DATABASE
PATHNAME OF PROJECT DATABASE:_____
_____ DATA LOADED INTO GIS

ATTACHMENT 5

SAMPLE TRACKING DATABASE EXAMPLE

CTO 020 SDG U06972

07/15/98

Proj Name	Job No	Sdg	Sample Number	Lab Id	Fraction	Sort	Lab Rec	B&R Rec	Turn-Time	WO No	Laboratory
PARRIS ISLAND	7394	U06972	PAI-03-SW-010	9806G972-001	LV	LV	06/03/98	07/14/98	41	9806G972	RECRA
PARRIS ISLAND	7394	U06972	PAI-03-SW-010	9806G972-001	MISC	CN	06/03/98	07/14/98	41	9806G972	RECRA
PARRIS ISLAND	7394	U06972	PAI-03-SW-010	9806G972-001	MISC	CR6	06/03/98	07/14/98	41	9806G972	RECRA
PARRIS ISLAND	7394	U06972	PAI-03-SW-010	9806G972-001	OS	OS	06/03/98	07/14/98	41	9806G972	RECRA
PARRIS ISLAND	7394	U06972	PAI-03-SW-010	9806G972-001	PAH	PAH	06/03/98	07/14/98	41	9806G972	RECRA
PARRIS ISLAND	7394	U06972	PAI-03-SW-010	9806G972-001	PEST/PCB	PCB	06/03/98	07/14/98	41	9806G972	RECRA
PARRIS ISLAND	7394	U06972	PAI-03-SW-010	9806G972-001	PEST/PCB	PEST	06/03/98	07/14/98	41	9806G972	RECRA

ATTACHMENT 6

EXAMPLE SAMPLE JAR LABELS

Tetra Tech NUS, Inc.
661 Andersen Drive
Pittsburgh, 15220
(412)921-7090

Project: NWS CHARLESTON
Location: 14SB01

Sample No: 14-P-001-01 | Matrix: SOIL
Date: | Time: | Preserve: 4° C
Analysis: TCL Volatiles
Sampled by: | Laboratory: RECRA

Tetra Tech NUS, Inc.
661 Andersen Drive
Pittsburgh, 15220
(412)921-7090

Project: NWS CHARLESTON
Location:

Sample No: | Matrix:
Date: | Time: | Preserve:
Analysis:
Sampled by: | Laboratory:

Tetra Tech NUS, Inc.
661 Andersen Drive
Pittsburgh, 15220
(412)921-7090

Project: NWS CHARLESTON
Location: 14SB01

Sample No: 14-P-001-01 | Matrix: SOIL
Date: | Time: | Preserve: 4° C
Analysis: TCL Semivolatiles, TAL Metals, Cyanide
Sampled by: | Laboratory: RECRA

Tetra Tech NUS, Inc.
661 Andersen Drive
Pittsburgh, 15220
(412)921-7090

Project: NWS CHARLESTON
Location:

Sample No: | Matrix:
Date: | Time: | Preserve:
Analysis:
Sampled by: | Laboratory:

Tetra Tech NUS, Inc.
661 Andersen Drive
Pittsburgh, 15220
(412)921-7090

Project: NWS CHARLESTON
Location: 14SB01

Sample No: 14-P-001-01 | Matrix: SOIL
Date: | Time: | Preserve: 4° C
Analysis: OTTO Fuel
Sampled by: | Laboratory: GEL

Tetra Tech NUS, Inc.
661 Andersen Drive
Pittsburgh, 15220
(412)921-7090

Project: NWS CHARLESTON
Location:

Sample No: | Matrix:
Date: | Time: | Preserve:
Analysis:
Sampled by: | Laboratory:

Tetra Tech NUS, Inc.
661 Andersen Drive
Pittsburgh, 15220
(412)921-7090

Project: NWS CHARLESTON
Location:

Sample No: | Matrix:
Date: | Time: | Preserve:
Analysis:
Sampled by: | Laboratory:

Tetra Tech NUS, Inc.
661 Andersen Drive
Pittsburgh, 15220
(412)921-7090

Project: NWS CHARLESTON
Location:

Sample No: | Matrix:
Date: | Time: | Preserve:
Analysis:
Sampled by: | Laboratory:

Tetra Tech NUS, Inc.
661 Andersen Drive
Pittsburgh, 15220
(412)921-7090

Project: NWS CHARLESTON
Location:

Sample No: | Matrix:
Date: | Time: | Preserve:
Analysis:
Sampled by: | Laboratory:

Tetra Tech NUS, Inc.
661 Andersen Drive
Pittsburgh, 15220
(412)921-7090

Project: NWS CHARLESTON
Location:

Sample No: | Matrix:
Date: | Time: | Preserve:
Analysis:
Sampled by: | Laboratory:

ATTACHMENT 7

ELECTRONIC DATA DELIVERABLE REQUIREMENTS FOR ANALYTICAL LABORATORIES

ELECTRONIC DATA FORMAT REQUIREMENTS

1.0 INTRODUCTION

The laboratory is to provide 3.5" high density diskette(s) containing separate database (DBF) files in the format specified in this Attachment. The electronic deliverable includes all environmental samples, sample dilutions, sample reanalyses, and laboratory quality control samples. **All entries in the electronic deliverable must agree exactly with the final entries reported on the hardcopy data package sample result summaries.** Any corrections made to the hardcopy data must also be made to the electronic file. Appropriate qualifiers as identified by the analytical protocol must also be designated; laboratory QC non-compliance codes are not to be depicted.

Each diskette is to be properly labeled with the laboratory name, project name, file name(s), and laboratory point of contact. Electronic files should be delivered in the same fashion as are the hard copy data packages. A separate .dbf file shall be made for each analytical fraction (by method) and each sample delivery group (SDG). The files shall be named with the first character being the analytical fraction designator, followed by an underscore, followed by the SDG name. For example, the file for the volatile fraction for SDG BR001 should be named V_BR001.DBF. Additionally, the laboratory must provide a hardcopy listing all electronic files saved to the diskette, indicating what analytical fraction and matrix the file data contained therein pertain to. All electronic data deliverables are due within the same time established for the associated hardcopy data packages.

In addition, the laboratory QC officer must read and sign a copy of the Quality Assurance Review Form displayed on the next page of this Attachment. Electronic deliverables are not considered to be complete without the accompanying Quality Assurance Review Form.

I _____, as the designated Quality Assurance Officer, hereby attest that all electronic deliverables have been thoroughly reviewed and are in agreement with the associated hardcopy data. The enclosed electronic files have been reviewed for accuracy (including significant figures), completeness and format. The laboratory will be responsible for any labor time necessary to correct enclosed electronic deliverables that have been found to be in error. I can be reached at (_____)_____if there are any questions or problems with the enclosed electronic deliverables.

Signature:_____ Title: _____ Date: _____

Revision 4
KAC 11-11-97

The analytical data shall be delivered electronically in a Dbase III file format (filename.dbf). The exact structure of the database is described in the table below. It shall be the responsibility of the laboratory to ensure that all electronic entries are in strict accordance with the information provided on the Form I.

An example database shall be sent for review prior to the first electronic deliverable in Dbase III format. The example file will be examined for completeness and comments will be sent to the laboratory. Any questions regarding the electronic deliverable shall be directed to Patrick Hooper at Brown and Root Environmental (412)921-8250.

DATA FIELD	DATA TYPE	FIELD WIDTH	DATA FIELD DESCRIPTION
SAMPLE_NO	C	25	Field sample ID as listed on the chain-of-custody. The sample number indicated in this field should never be truncated. The only exception for this field not matching the chain-of-custody is for reanalyses and matrix spike results in which a RE or MS suffix will be added to the sample number respectively.
TRUNCATE	C	15	If the field sample ID listed on the Chain of Custody is truncated by the laboratory for use with the laboratory software, the truncated sample ID should appear in this field.
LAB_ID	C	15	Laboratory number for the given sample.
LABORATORY	C	25	Laboratory name.
BATCH_NO	C	10	Laboratory code for batch of samples included in a given run.
ASSOC_BLNK	C	15	Laboratory name of the method blank associated with that particular batch of samples.
QC_TYPE	C	15	Normal Environmental Sample = "NORMAL", Laboratory Duplicate = "DUPLICATE", Matrix Spike = "MS", Matrix Spike Duplicate = "MSD", Laboratory Control Sample = "LCS", Laboratory Control Sample Duplicate = "LCSD", Method Blank = "M_BLANK", Preparation Blank = "P_BLANK".
SAMP_DATE	D	8	Date of sample collection as indicated on the Chain of Custody. Example: 11/07/93.
REC_DATE	D	8	Date sample was received by the laboratory.
EXTR_DATE	D	8	Date sample was extracted or prepared by the laboratory.
ANAL_DATE	D	8	Date sample was analyzed by the laboratory.
RUN_NUMBER	N	2 (0)	The number of the analytical run for a given sample in sequence. For example, if a sample is diluted and reanalyzed, the original run number would be 1 and the reanalysis would be 2.
SDG	C	15	Sample delivery group identifier assigned by the laboratory. This number should exactly match the SDG designated on the hardcopy data package.

DATA FIELD	DATA TYPE	FIELD WIDTH	DATA FIELD DESCRIPTION
PROJECT_NO	C	10	Identification of Project Number or CLEAN Task Order (CTO) number.
PROJ_MNGR	C	25	The Brown & Root Project Manager's last name, followed by a comma, followed by the first initial of the Project Manager (e.g. Hutson, D).
PARAMETER	C	45	Chemical or analyte name exactly as reported on Form I.
CAS_NO	C	10	Chemical Abstract Service number for the parameter listed. The CAS number should be reported exactly as it is listed in publications such as the Merck Index. This field should be left blank for those parameters not having CAS numbers (e.g. Total Organic Carbon).
FRACTION	C	5	Metals = 'M', Volatiles = 'OV', Semivolatiles/BNAs = 'OS', Pesticides = 'PEST', Herbicides = 'HERB', Polychlorinated Biphenyls = 'PCB', Explosives = 'EXP', Any petroleum hydrocarbon or fuel = 'TPH', Wet Chemistry = 'WET', Radionuclide = 'RAD', Miscellaneous = 'MISC'
METHOD	C	20	Analytical method used to quantitate parameter concentrations as listed in the laboratory technical specification (e.g. '8270A' for SW-846 Method 8270A.
LAB_RESULT	N	20 (6)	Reported value in units specified in the UNITS field containing the proper number of significant digits. The % Recovery shall be placed in this field for matrix spike and laboratory control sample results.
UNITS	C	5	The units of measure as reported on the Form I.
LAB_QUAL	C	2	The laboratory qualifier as reported on the Form I. For example, a 'U' qualifier should be used for all nondetected results.
IDL	N	15 (6)	Instrument detection limit in units specified in the UNITS field.
MDL	N	15 (6)	Method detection limit in units specified in the UNITS field and method specified in the METHOD field.
CRDL_CRQL	N	15 (6)	Contract Required Detection/Quantitation Limit in the units specified in the UNITS field. RDL for non-CLP parameters.
DIL_FACTOR	N	6 (1)	Dilution factor.
PCT_MOIST	N	5 (1)	Percent moisture for soil samples; blank for water samples.
COMMENTS	C	20	Analytical result qualifier or comment other than that listed in the LAB_QUAL field. Example: 'Reanalysis'.

C = Character string (everything shall be reported in capital letters)
N = Numeric string (decimal places are in parentheses in field width column)
D = Date (Ex: 05/25/97)

ATTACHMENT 8

DATABASE STRUCTURE

Database Structure

The NAS Dallas master database shall contain 18 standard tables to store all chemical, geological, and hydrogeological data. The structure, indexes, primary keys, and relations for each table are defined below.

TABLE: well
PRIMARY KEY: location

Table Structure

FIELD	TYPE	DESCRIPTION
Location	C (25)	Unique location name.
post_id	C (20)	Location name as derived from original source document.
instal_date	D (8)	Date the monitoring well was installed. Null for other location types.
loc_type	C (4)	Type of location.
Northing	N (15,4)	Northing coordinate in horizontal datum referenced in the HORIZ_DATUM field.
Easting	N (15,4)	Easting coordinate in horizontal datum referenced in the HORIZ_DATUM field.
horiz_datum	C (8)	Datum in which the horizontal coordinates were derived.
grnd_surf	N (15,4)	Ground surface elevation with reference to mean sea level in vertical datum referenced in the VERT_DATUM field
vert_datum	C (25)	Datum in which the vertical coordinates were derived.
datum_state	C (2)	State for which datum was developed
Surveyed	L (1)	Logical field denoting whether positional data were surveyed or digitized.
Surveyor	C (50)	Company who performed the survey.
survey_date	D (8)	Date in which survey was performed.
surv_method	C (25)	Surveying method used.
Longitude	N (15,4)	Longitude
Latitude	N (15,4)	Latitude

Table Indexes

INDEX	TYPE
Location	Primary
Hd	Regular
Vd	Regular
loc_type	Regular

Table Relations:

Relation 1
*RelatedChild loc_type
*RelatedTable loc_type_vvl
*RelatedTag loc_type

Relation 2
*RelatedChild hd
*RelatedTable horiz_datum_vvl
*RelatedTag hd

Relation 3
*RelatedChild vd
*RelatedTable vert_datum_vvl
*RelatedTag vd

TABLE: loc_type_vvl - Valid value list for LOC_TYPE field in the well table.
PRIMARY KEY: loc_type

Table Structure

FIELD	TYPE	DESCRIPTION
loc_type	C (4)	Location type
Description	C (40)	Description of location type

Table Indexes

INDEX	TYPE
loc_type	Primary

TABLE: horiz_datum_vvl - Valid value list for HORIZ_DATUM field in the well table.
PRIMARY KEY: hd (horiz_datum)

Table Structure

FIELD	TYPE	DESCRIPTION
Horiz_datum	C (15)	Datum in which x,y coordinates reflect

Table Indexes

INDEX	TYPE
hd (horiz_datum)	Primary

Table Indexes

INDEX	TYPE
loc_type	Primary

TABLE: vert_datum_vvl - Valid value list for VERT_DATUM field in the well table.
PRIMARY KEY: vd (vert_datum)

Table Structure

FIELD	TYPE	DESCRIPTION
Vert_datum	C (15)	Datum in which z coordinate reflects

Table Indexes

INDEX	TYPE
vd (vert_datum)	Primary

TABLE: sample_data - Sample data table
PRIMARY KEY: nsample

Table Structure

FIELD	TYPE	DESCRIPTION
Location	C (25)	Unique location name.
Matrix	C (4)	Sample matrix
Nsample	C (35)	Unique sample identification
Sample	C (25)	Sample identification as designated on Chain-of-Custody
Sacode	C (8)	Sample code for reference to field duplicates
top_depth	N (5,1)	Depth in feet to the top of the sample interval. Applicable for soil and sediment samples.
Bottom_depth	N (5,1)	Depth in feet to the bottom of the sample interval. Applicable for soil and sediment samples. *Rule Expression*: if(bottom_depth>0.top_depth<=bottom_depth)
qc_type	C (2)	Quality control type

Status	C (10)	Status of sample location – Normal or excavated
sample_date	D (8)	Date in which sample was collected
Validated	L (1)	Logical field denoting whether or not data validation was performed on sample
coll_method	C (10)	Sample collection method
cto_proj	C (5)	Clean task order (Navy) or project number in which the sample was collected (e.g. "129")
proj_manager	C (25)	Internal projcect manager for which the data was originally generated (e.g. "Hutson. D.").

Table Indexes

INDEX	TYPE
Location	Regular
Nsample	Primary
Sacode	Regular
Matrix	Regular
Status	Regular
qc_type	Regular
coll_meth	Regular

Table Relations:

Relation 1
*RelatedChild sacode
*RelatedTable sacode_vvl
*RelatedTag sacode

Relation 2
*RelatedChild qc_type
*RelatedTable qc_type_vvl
*RelatedTag qc_type

Relation 3
*RelatedChild matrix
*RelatedTable matrix_vvl
*RelatedTag matrix

Relation 4
*RelatedChild location
*RelatedTable well
*RelatedTag location

Relation 5
*RelatedChild coll_meth
*RelatedTable coll_method_vvl
*RelatedTag coll_meth

TABLE: sacode_vvl - Sample code valid value list for SACODE field in sample_data.dbf
PRIMARY KEY: sacode

Table Structure

FIELD	TYPE	DESCRIPTION
Sacode	C (8)	Sample code designating whether sample is a normal environmental sample, a field duplicate or the average of field duplicate pairs
Description	C (30)	Description of sacode entry

Table Indexes

INDEX	TYPE
Sacode	Primary

TABLE: qc_type_vvl - Quality control valid value list for QC_TYPE field in sample_data.dbf
PRIMARY KEY: qc_type

Table Structure

FIELD	TYPE	DESCRIPTION
qc_type	C (10)	Quality control type
Description	C (30)	Description of quality control type

Table Indexes

INDEX	TYPE
qc_type	Primary

TABLE: matrix_vvl - Matrix valid value list for MATRIX field in sample_data.dbf
PRIMARY KEY: matrix

Table Structure

FIELD	TYPE	DESCRIPTION
Matrix	C (4)	Sample matrix
Description	C (25)	Description of sample matrix code

Table Indexes

INDEX	TYPE
Matrix	Primary

TABLE: well_completion
PRIMARY KEY: None

Table Structure

FIELD	TYPE	DESCRIPTION
Location	C (25)	Unique location name
top_casing	N (8,2)	Elevation of top of well casing in vertical datum found in VERT_DATUM in the well table
hole_diameter	N (5,1)	Diameter of the drilled hole in inches *Rule Expression*: hole_diameter>casing_id.AND.hole_diameter>casing_od
scr_aquifer	C (30)	Aquifer name in which the screen resides
screen_material	C (15)	Type of material in which the screen is constructed from
scrn_slot_size	N (5,1)	Screen slot size in thousandths of an inch
scrn_top_depth	N (5,2)	Depth below ground surface to the top of the screen (in feet)
scrn_bot_depth	N(5,2)	Depth below ground surface to the bottom of the screen *Rule Expression*: if(scrn_bot_depth>0,scrn_top_depth<scrn_bot_depth)
scrn_top_elev	N (6,2)	Elevation the top of the screen in vertical datum found in VERT_DATUM in the well table.
scrn_bot_elev	N (6,2)	Elevation the top of the screen in vertical datum found in VERT_DATUM in the well table. *Rule Expression*: if(scrn_bot_elev>0,scrn_top_depth>scrn_bot_depth)
drill_method	C (15)	Drilling method for well installation
Contractor	C (20)	Drilling contractor
casing_material	C (15)	Type of material in which the casing is constructed from
depth_to_seal	N (8,2)	Depth below ground surface to seal (in feet)

seal_material	C (15)	Type of material in which the seal is constructed from
fill_top_depth	N (6,2)	Depth below ground surface to the top of fill material (in feet)
fill_bot_depth	N (6,2)	Depth below ground surface to the bottom of fill material (in feet). *Rule Expression*: if(fill_bot_depth>0,fill_top_depth<scrn_bot_depth)
fill_material	C (20)	Type of material used for fill.
comments	M (4)	Geologist's comments

Table Indexes

INDEX	TYPE
location	Regular

Table Relations:
Relation 1
*RelatedChild location
*RelatedTable well
*RelatedTag location

TABLE: lithology
PRIMARY KEY: None

FIELD	TYPE	DESCRIPTION
location	C (25)	Unique location name
top_lithology	N (6,2)	Depth in feet below ground surface to the top of lithologic unit
bottom_lithology	N (6,2)	Depth in feet below ground surface to the bottom of lithologic unit
uscs_code	C (5)	Unified Soil Classification Service Code for lithology type
blow_counts	C (8)	Number of blow counts recorded on boring log
description	C (80)	Geologist's description of lithology
comments	M (4)	Geologist's comments.

Table Indexes

INDEX	TYPE
location	Regular
uscs_code	Regular

Table Relations:
Relation 1
*RelatedChild location
*RelatedTable well
*RelatedTag location
Relation 2
*RelatedChild uscs_code
*RelatedTable lithology_vvl
*RelatedTag uscs_code

TABLE: lithology_vvl - Lithology valid value list for USCS_CODE field in lithology.dbf
PRIMARY KEY: uscs_code

Table Structure

FIELD	TYPE	DESCRIPTION
uscs_code	C (4)	Unified Soil Classification Service Code for lithology type
descript	C (70)	Description of lithology for given USCS code

Table Indexes

INDEX	TYPE
uscs_code	Primary

TABLE: coll_method_vvl - Collection method valid value list for COLL_METHOD field in sample_data.dbf
PRIMARY KEY: coll_meth

Table Structure

FIELD	TYPE	DESCRIPTION
coll_method	C (10)	Sample collection method

Table Indexes

INDEX	TYPE
coll_method	Primary

TABLE: cas_vvl - CAS number valid value list for CAS field in analytical results.dbf
PRIMARY KEY: cas

Table Structure

FIELD	TYPE	DESCRIPTION
parameter	C (40)	Parameter or chemical name
cas	C (11)	Chemical Abstracts Service Number

Table Indexes

INDEX	TYPE
parameter	Regular
cas	Primary

TABLE: analytic_results
PRIMARY KEY: nfp (nsample+fraction+parameter)

Table Structure

FIELD	TYPE	DESCRIPTION
nsample	C (35)	Unique sample identification
lab_id	C (15)	Laboratory sample identification
laboratory	C (25)	Laboratory name
batch_no	C (10)	Analytical batch number
assoc_blnk	C (15)	Associated blank
extr_date	D (8)	Extraction date
anal_date	D (8)	Analysis date
run_number	I (4)	Sequential analytical run number
sdg	C (15)	Sample delivery group
parameter	C (45)	Parameter or chemical name (using IUPAC nomenclature where appropriate)
cas	C (11)	Chemical Abstracts Service Number
fraction	C (5)	Analtytical fraction
method	C (20)	Analytical method
lab_result	N (20,6)	Analytical result as reported by the laboratory
lab_qual	C (5)	Qualifier as reported by the laboratory
val_res	N (20,6)	Final result (via validation or otherwise)

result	C (20)	Final analytical result with the correct number of significant figures
val_qual	C (3)	Validation qualifer (null if data were not validated)
qual	C (3)	Final qualifer (validation or otherwise)
units	C (5)	Units of measure for the RESULT field
idl	N (15,6)	Instrument detection limit (same units as UNITS field)
mdl	N (15,6)	Method detection limit (same units as UNITS field)
crdl_crql	N (15,6)	Contract required detection/quantitation limit (same units as UNITS field)
dil_factor	N (6,1)	Dilution factor
pct_moist	N (5,1)	Percent moisture
comments	C (20)	Comments from laboratory analyst

Table Indexes

INDEX	TYPE
nfp	Primary
units	Regular
qual	Regular
fraction	Regular
parameter	Regular
nsample	Regular
cas	Regular

Table Relations:

<u>Relation 1</u>
*RelatedChild cas
*RelatedTable cas_vvl
*RelatedTag cas
<u>Relation 2</u>
*RelatedChild units
*RelatedTable units_vvl
*RelatedTag units
<u>Relation 3</u>
*RelatedChild qual
*RelatedTable qual_vvl
*RelatedTag qual
<u>Relation 4</u>
*RelatedChild fraction
*RelatedTable fraction_vvl
*RelatedTag fraction
<u>Relation 5</u>
*RelatedChild parameter
*RelatedTable para_vvl
*RelatedTag para
<u>Relation 6</u>
*RelatedChild nsample
*RelatedTable sample_data
*RelatedTag nsample

TABLE: units_vvl - Units valid value list for UNITS field in analytical_results.dbf
PRIMARY KEY: Units

Table Structure

FIELD	TYPE	DESCRIPTION
Units	C (8)	Units of measure for chemical analysis
Description	C (20)	Description of units

Table Indexes

INDEX	TYPE
Units	Primary

TABLE: qual_vvl
PRIMARY KEY: qual - Qualifier valid value list for QUAL field in analtyic_results.dbf

Table Structure

FIELD	TYPE	DESCRIPTION
Qual	C (5)	Final QA qualifier
Description	C (60)	Definition of qualifier

Table Indexes

INDEX	TYPE
Qual	Primary

TABLE: fraction_vvl - Analytical fraction valid value list for FRACTION field in analytic_results.dbf
PRIMARY KEY: fraction

Table Structure

FIELD	TYPE	DESCRIPTION
Fraction	C (10)	Analytical fraction
Description	C (35)	Description of fraction

Table Indexes

INDEX	TYPE
Fraction	Primary

TABLE: para_vvl
PRIMARY KEY: parameter

Table Structure

FIELD	TYPE	DESCRIPTION
Para	C (60)	Parameter or chemical name
frac_name	C (35)	Analytical fraction for given parameter

Table Indexes

INDEX	TYPE
Para	Primary

TABLE: fluid
PRIMARY KEY: None

Table Structure

FIELD	TYPE	DESCRIPTION
Location	C (25)	Unique location name
Fluid_date	D (8)	Date measurement was taken
meas_elev	N (8,2)	Measuring point elevation
dep_to_water	N (6,2)	Depth below ground surface to water table (in feet)
dep_to_fp	N (6,2)	Depth below ground surface to free product (in feet)
elev_water	N (8,2)	Elevation of water level
elev_fp	N (8,2)	Elevation of free product
prod_thick	N (6,2)	Product thickness in feet

Table Indexes

INDEX	TYPE
location	Regular

Table Relations:
Relation 1
*RelatedChild location
*RelatedTable well
*RelatedTag location

ATTACHMENT 9

ARCVIEW GIS STRUCTURE

ArcView GIS Structure

The NAS Dallas ArcView GIS shall have the following directory structure and database table structure.

Part One: Directory Structure

The following table defines the directory structure and major file names/types located within each directory.

Main subdirectory	First tier subdirectories	Second tier subdirectories	Files/Types
p:\gis\project name\	database\		coordinate.dbf cross_reference.dbf res_ gw.dbf res_so.dbf res_sd.dbf res_sw.dbf well_completion.dbf
		criteria\	crit_ gw.dbf crit_so.dbf crit_ sd.dbf crit_sw.dbf crit_des.dbf
	mapping\	aerial\	registered aerial photos
		drg\	USGS Digital Raster Graphic
		image\	GeoStatistic Layers, pictures of sites, equipment, EVS, and all other raster files.
		dwg\	AutoCAD files
		dgn\	Microstation files
		shp\	samp_gw.shp .dbf .shx samp_so.shp .dbf .shx samp_sd.shp .dbf .shx samp_sw.shp .dbf .shx and all other AV shape files
	working\	database\	files used to generate specific drawings will be put under the working subdirectory in subdirectories similar to database & mapping. These will not be included in CD deliverable.
		mapping\	same as above

Part Two: Database Table Structure

The ArcView GIS will contain separate database tables to store analytical, criteria, and coordinate information. The structure of these tables is presented below.

Analyticl Data Table

The following table lists all the fields contained in the analytic database table.

FIELD	VISIBLE	ALIAS	DESCRIPTION
site	Yes	Site or SWMU	Site or SWMU
location	Yes	Location	Unique location name
nsample	Yes	Sample	Unique sample identification
sample	No		Sample identification as designated on Chain-of-Custody
sample_date	Yes	Sample Date	Date in which sample was collected
matrix	Yes	Matrix	Sample matrix
sacode	Yes	Sample Code	Sample code for reference to field duplicates
depth	Yes	Depth	Depth in feet to the middle of the sample interval. Applicable for soil and sediment samples.
top_depth	Yes	Top Depth	Depth in feet to the top of the sample interval. Applicable for soil and sediment samples.
bottom_depth	Yes	Bottom Depth	Depth in feet to the bottom of the sample interval. Applicable for soil and sediment samples. Rule Expression: if(bottom_depth>0,top_depth<=bottom_depth)
parameter	Yes	Parameter	Parameter or chemical name (using IUPAC nomenclature where appropriate)
cas	Yes	CAS	Chemical Abstracts Service Number
fraction	Yes	Fraction	Analytical fraction
val_res	Yes	Numeric Result	Final result (via validation or otherwise)
qual	Yes	Qualifier	Final qualifier (validation or otherwise)
units	Yes	Units	Units of measure for the RESULT field
method	Yes	Method	Analytical method
status	Yes	Status	Status of sample location – Normal or excavated
validated	Yes	Validated	Logical field denoting whether or not data validation was performed on sample
coll_method	Yes	Collection Method	Sample collection method
cto_proj	Yes	CTO	Clean task order (Navy) or project number in which the sample was collected (e.g. "129")
proj_manager	Yes	Project Manager	Internal project manager for which the data was originally generated (e.g. "Hooper, P.").
lab_id	No	Laboratory ID	Laboratory sample identification
laboratory	No	Laboratory	Laboratory name
batch_no	No	Batch Number	Analytical batch number

assoc_blnk	No	Associated Blank	Associated blank
extr_date	No	Extraction Date	Extraction date
anal_date	No	Analysis date	Analysis date
run_number	No	Run Number	Sequential analytical run number
sdg	No	SDG	Sample delivery group
lab_result	No	Result	Analytical result as reported by the laboratory
lab_qual	No	Lab Qualifier	Qualifier as reported by the laboratory
result	No	String Result	Final analytical result with the correct number of significant figures
val_qual	No	Validation Qualifier	Validation qualifier (null if data were not validated)
idl	No	Detection Limit	Instrument detection limit (same units as UNITS field)
mdl	No	Detection Units	Method detection limit (same units as UNITS field)
crdl_crql	No		Contract required detection/quantitation limit (same units as UNITS field)
dil_factor	No	Dilution factor	Dilution factor
pct_moist	No	Percent moisture	Percent moisture
ourresult	No		
qc_type	No		Quality control type
comments	No	Comments	Comments from laboratory analyst

Criteria Table

Each medium will have a criteria table to specify the applicable criteria for all parameters.

FIELD	ALIAS	DESCRIPTION
parameter	Parameter	Parameter or chemical name (using IUPAC nomenclature where appropriate)
epa_mcl	None	Federal MCL – groundwater

Note: usually there will be many criteria fields. This example table only shows the "epa_mcl" criteria field.

Criteria Description Table

This table stores the definition or description of all standards and criteria used in the project. For example, epa_mcl's media would be GW, description would be "Federal Maximum Contaminant Level".

FIELD	Visible	DESCRIPTION
Field	Yes	
Media	Yes	
Descript	Yes	

Coordinate Table

The coordinate table holds all the geographic position information of sampling locations

FIELD	Visible	ALIAS	DESCRIPTION
Location	Yes	None	Unique location name.
post_id	Yes	Location Designation	Location name as derived from original source document.
instal_date	No	Installation Date	Date the monitoring well was installed. Null for other location types.
loc_type	Yes	Location Type	Type of location. Example MW, HP, etc.
northing	Yes		Northing coordinate in horizontal datum referenced in the HORIZ_DATUM field.
Easting	Yes		Easting coordinate in horizontal datum referenced in the HORIZ_DATUM field.
Grnd_surf	Yes	Ground Surface Elevation	Ground surface elevation with reference to mean sea level in vertical datum referenced in the VERT_DATUM field
horiz_datum	Yes	Horizontal Datum	Datum in which the horizontal coordinates were derived.
Vert_datum	Yes	Vertical Datum	Datum in which the vertical coordinates were derived.
Datum_state	Yes	Coordinate System	State for which datum was developed
Surveyed	Yes		Logical field denoting whether positional data were surveyed or digitized.
Surveyor	Yes		Company who performed the survey.
Survey_date	No	Survey Date	Date in which survey was performed.
Surv_method	No	Survey Method	Surveying method used.
Longitude	No		Longitude
Latitude	No		Latitude
gw_code	Yes		This will be populated by database personnel. It will be used for event driven theme.
Sd_code	Yes		This will be populated by database personnel. It will be used for event driven theme.
So_code	Yes		This will be populated by database personnel. It will be used for event driven theme.
Sw_code	Yes		This will be populated by database personnel. It will be used for event driven theme.
_nullflags	No		Various fields are put in by database starting here and followed by several fields. Make all of these invisible

APPENDIX C

BASE-WIDE BACKGROUND SOIL INVESTIGATION DATA MANAGEMENT PLAN FOR NSWC CRANE

1.0 PROJECT PLANNING

A large amount of environmental and physical data has been collected in support of the Installation Restoration (IR) and Underground Storage Tank (UST) programs. TtNUS has the responsibility of managing this data in a basewide relational database and GIS. The contents of the database shall be outlined in the Sitewide Data Catalog (which at a minimum, contains the data fields identified in Attachment 1). The Data Catalog shall outline what data is contained within the database (by investigation, media, etc.), the generator of the data (TtNUS, Corps of Engineers, etc.), and the level of quality of the data where applicable. It should be noted whether or not the analytical data were validated and to what level. It is the responsibility of the TtNUS data manager to coordinate with the NSWC Crane project team in order to keep the Data Catalog current and make available the most recent version to all team members. A copy of the Data Catalog shall be maintained in the project central file at the office of TtNUS. It is the responsibility of the all team members to ensure that the Data Catalog is correct and current and shall notify the TtNUS data manager of any newly generated data that will support the needs of the project.

Prior to every data collection event, the TOM shall call a kick-off meeting to outline the data needs of the task order and to review the data flow process (Attachment 2). Attendees to the kick-off meeting should include the TOM, the Human Health Risk Assessment (HHRA) lead, the Field Operations Leader (FOL), the project chemist, the data management lead and the Geographic Information System (GIS) lead. The data management lead shall distribute a copy of the database checklist (Attachment 3) and shall lead the project team through its contents. The database checklist will allow the project team to determine how the data will be managed and manipulated in order to achieve the project needs and objectives. A completed copy of the database checklist shall be maintained in the project central file and distributed to all members of the project team within seven days of the kick-off meeting.

2.0 NEWLY GENERATED DATA

Upon directive from SOUTHDIV to collect additional site data, the TOM shall coordinate with the designated data management lead and GIS lead for the project. It is the responsibility of the FOL to comply with the sample and location nomenclature outlined in the Work Plan. It is also the responsibility of the FOL to coordinate with the GIS lead to ensure that all survey technical specifications require the proper coordinate system, which is Indiana State Planar - North American Datum 1983 for the horizontal coordinates and National Geodetic Vertical Datum 1988 for the vertical coordinates.

Prior to field mobilization, the FOL shall coordinate with the Sample Management Coordinator (SMC) to initiate a sample tracking process. It is the responsibility of the TOM to ensure that a sampling tracking procedure is implemented. Sample Tracking Request Forms, a sample tracking database example, and example jar labels are included as Attachments 4, 5 and 6, respectively. In the event that a field change has taken place, the FOL is required to complete the Field Change Notification Form that will be forwarded to all members of the project team.

According to all laboratory technical specifications for NSWC Crane, the analytical laboratories will be contractually required to deliver the analytical data in NSWC

Crane standard Electronic Data Deliverable (EDD) format (Attachment 7). Particular attention should be paid to the EDD requirements for validated vs. non-valididated data. Once all samples and analyses have been accounted for, the SMC shall forward the analytical data to TtNUS for incorporation into the NSWC Crane database which is located on the Local Area Network (LAN) in Pittsburgh, PA. The NSWC Crane database structure is presented in Attachment 8.

3.0 HISTORICAL DATA

In the event that the NSWC Crane project team decides that existing hardcopy data not outlined in the Data Catalog (Attachment 1) needs to be incorporated into the project database, SOUTHDIV shall provide directive to the appropriate consultant to incorporate the data into the project database. The data management lead shall review the hardcopy data and prepare a summary of the samples and analyses that need to be entered. The format of the summary table should be similar to the sample tracking database provided in Attachment 5. It is the responsibility of the TOM to review the sample summary table and verify that the entry of this data will satisfy the project requirements. The data management lead shall physically edit the hardcopy analytical data to clearly designate which information on the hardcopy needs to be entered into the database. Copies of the marked-up data must be distributed to two separate parties for entry into an Excel spreadsheet. Upon completion of the dual-key entry, the data management lead shall electronically compare the two data files to identify discrepancies and correct the data appropriately. The database should then be queried against the sample summary table to ensure that all pertinent data has been entered and checked for accuracy.

The data management lead shall coordinate with the GIS lead to acquire the sample location data (Attachment 8) for those samples that need to be entered. Sample location maps should be used to digitize the sample locations using the base mapping layer in the GIS. To the extent possible, the GIS lead shall capture, as metadata, the accuracy of the sample location maps used to digitize the location

coordinates. If no sample location maps or other positional information exist for the historical data, the project team should evaluate the utility of this data in the NSWC Crane database.

4.0 MAPPING AND GRAPHICS

CADD mapping is generally provided by the activity. We currently do not use metadata to track changes to the mapping. In addition, Tri-Service Spatial Data Standards (TSSDS) are not utilized unless the mapping from the base already incorporates them. TSSDS also is not used in the final GIS, based on the view that limited utility is gained from the substantial time required to incorporate the standards.

In addition to CADD mapping, Digital Ortho Quarter (DOQ) Quads, Aerial Photography, and USGS 7.5 minute Quads are obtained. The Quads are obtained from either the USGS or other suppliers, while the aerial photography is provided by the activity. As necessary, the images are warped to the predetermined coordinate system using Microstation. Again, metadata are not used to track the changes. From survey data, sampling locations are organized, and then a sample-vs-location table is built so that the data can be loaded into the sample_data.dbf table (Attachment 8).

5.0 THE ENVIRONMENTAL GEOGRAPHIC INFORMATION SYSTEM (EGIS)

All environmental data collected in support of the NSWC Crane project shall be incorporated into the GIS. The themes, layers and database information contained in the GIS is outlined in the Data Catalog (Attachment 1). The NSWC Crane GIS shall be made available to all members of the project team. CD-ROM EGIS deliverables shall be made available upon request from SOUTHDIV.

6.0 ASSIMILATION OF DATA FROM OUTSIDE SOURCES

When environmental data is collected by a contractor other than TtNUS, it is the responsibility of the SOUTHDIV Remedial Project Manager (RPM) to notify the TtNUS TOM. The RPM should forward a scope of work directing TtNUS to coordinate with the contractor and incorporate their data into the basewide GIS. To the extent possible, the RPM should direct the Navy Contractor to supply the data to TtNUS in the format outlined in Attachment 8. Once TtNUS has incorporated the data into the GIS, a hardcopy report shall be sent to the contractor for verification that all pertinent data have been incorporated in a complete and accurate fashion.

7.0 SOFTWARE

TtNUS will standardize on the following software packages when managing and manipulating data for the NSWC Crane project:

- Data Management - Microsoft Visual FoxPro 6.0
- GIS - ArcView 3.1 (see Attachment 9 for instructions)
- Geostatistics (2-D Kriging) - Geosoft 3.1b
- 3-D Visualization - EVS Pro 3.0
- Ground Water Modeling - GMS
- Statistical Analysis - Statistica 5.1
- Terrain Analysis - TerraModel 9.4.1

8.0 STORAGE OF DATA

TtNUS utilizes NT for Networks as its Information Management System (IMS). The NT IMS has a storage capacity of 2 Gigabytes and currently serves over 110 desktop computers. The NT IMS automatically backs-up the system on a daily basis, thereby disallowing more than one day of work being lost should the network crash or malfunction. The database management and GIS groups have been allocated distinct drives on the Local Area Network (LAN). All environmental data for the NSWC Crane Project shall be stored on \\nusrpitbdc1\sdiv\NSWC_Crane subfolder of this drive on the NT Server. All tables, queries, programs and reports shall be saved in the NSWC_Crane.pjx file in Microsoft Visual FoxPro. The NSWC Crane EGIS shall be stored on \\nusrpitbdc1\gis\NSWC_Crane on the NT Server. All ArcView project files (*.apr) shall be documented in a text file called readme_project.txt. This text file shall also be stored on \\nusrpitbdc1\gis\NSWC_Crane.

ATTACHMENT 1

DATA CATALOG
(Minimum Requirements)

DATA CATALOG DATA FIELDS

NSWC CRANE

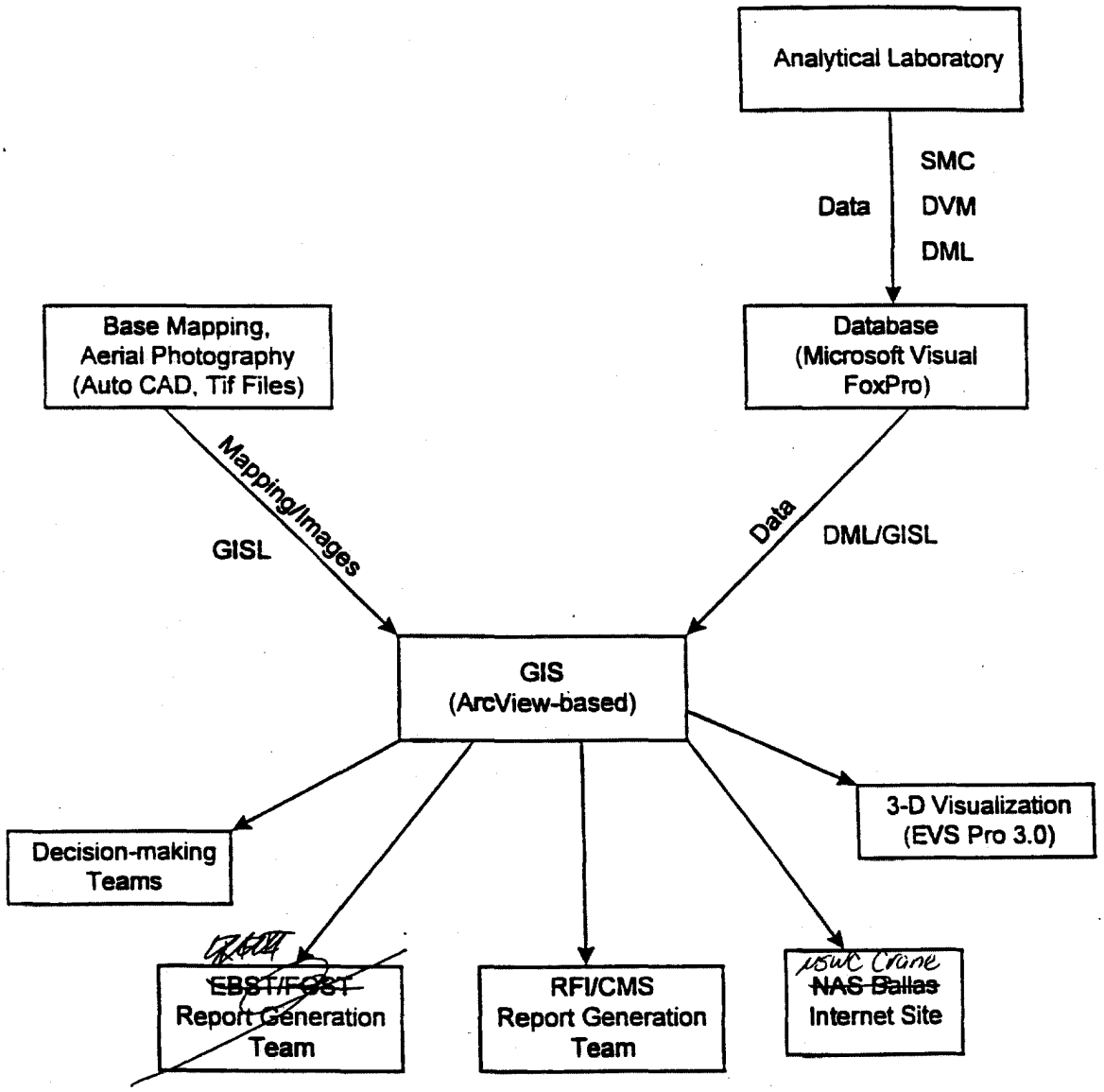
CRANE, INDIANA

Category	RFI Phase	Medium Sampled	Sample Type	No. of Samples	Fraction Analyzed

ATTACHMENT 2

THE DATA FLOW PROCESS

THE DATA FLOW PROCESS



Notes:

SMC = Sample Management Coordinator
DVM = Data Validation Manager
DML = Data Management Leader
GISL - GIS Leader

ATTACHMENT 3

DATABASE CHECKLIST

DATABASE PLANNING CHECKLIST

PROJECT NAME _____ PROJECT NUMBER _____

PROJECT MANAGER _____ PLANNING DATE _____

1 Provide a general description of the project (regulatory authority, media to be samples, approximate number of samples by media, analyses by media, data evaluation tasks required):

_____
_____
_____
_____
_____

2. Provide a general description of the sample nomenclature that will be used for samples collected by Brown & Root Environmental:

_____
_____
_____
_____

3. Will historical data be entered in the database? Yes No

4. Will historical data be used to define the nature and extent of contamination? Yes No

5. Will historical data be used for risk assessment purposes? Yes No

6. How much historical data exists (i.e., number of samples by matrix, analysis by matrix)?

_____
_____
_____
_____

7. In what format will the historical data be provided? Hardcopy Electronic

8. If historical data are in electronic form, what software was used and what is the format?

_____
_____
_____
_____

9. If historical data are in hardcopy form, will Form I's, summary tables, or reports be provided? Copies of historical data will be necessary to generate a budget estimate.

_____
_____
_____
_____

10. Will Quality Assurance review of historical data be necessary? Yes No

11. If Quality Assurance review of historical data is necessary, describe the scope of the Quality Assurance review:

_____
_____
_____
_____

12. Will a GIS database be necessary for the project? Yes No

13. What nomenclature has been (will be) used to identify field duplicate samples?

_____
_____
_____
_____

14 Will field duplicate results be averaged and presented as one result in the data base? Will they be presented as distinct results, or will both the average and the distinct results be presented?

_____
_____
_____
_____

15. How will the average value for duplicate samples be determined on a matrix-specific basis?

_____
_____
_____
_____

16. Are any unvalidated data to be included in the database? Yes No

17. Will unvalidated data be used for defining the nature and extent of contamination? Yes No

18. Will unvalidated data be used for risk assessment purposes? Yes No

19. Are any field screening data to be included in the database? Yes No

20. Will field screening data be used for defining the nature and extent of contamination? Yes No

21. Will field screening data be used for risk assessment purposes? Yes No

22. Will statistical correlation of laboratory and field screening data be necessary? Yes No

23. If a correlation exists between field screening and laboratory data, will the results of regression analysis be used to define nature and extent? Yes No

24. If a correlation exists between field screening and laboratory data, will the results of regression analysis be used to support the risk assessment? Yes No

25. Will field parameters be included in the database (e.g., pH, conductance, temperature)? Yes No

26. Will statistical correlations be necessary for TCLP versus RAS/SAS data? Yes No

27. Will statistical correlations be necessary for filtered versus unfiltered samples? Yes No

28. Will any other statistical correlations be necessary? Yes No

29. Are there wells that have been screened in different aquifers? Yes No

30. Will data for various aquifers be segregated by depth? Yes No

31. Can the sample nomenclature system be used to identify wells in different aquifers? Yes No

32. Will samples from other matrices (soil, sediment, or surface water) be segregated by depth? Yes No

33. Can the sample nomenclature system be used to identify depth-specificity? Yes No

34. Have any removal actions be performed at the site? Yes No

If removal actions have been performed, plan and cross-sectional views reflecting the extent of the removal action must be provided.

35. Will any composite sample results be included in the database? Yes No

36. If composite samples are included how will they be used for the nature and extent of contamination?

_____
_____
_____

37 If composite samples are included how will they be used for the risk assessment?

_____
_____
_____

38. Will the site be segregated into Areas of Concern, Solid Waste Management Units, etc? Yes No

39. Is the sample nomenclature adequate for such segregation? Yes No

If the sample nomenclature is inadequate for assigning samples to an AOC or SWMU, the Project Manager or designee must provide a base map of tabular summary clearly delineating the relationship between each sample and each AOC/SWMU.

40. Were any temporal samples collected (e.g., quarterly sampling of wells)? Yes No

41. If temporal samples were collected, how will they be used to define the nature and extent of contamination?

_____
_____
_____

42. If temporal samples were collected, how will they be used to support the risk assessment?

_____
_____
_____

43. Are State, Federal, or Regional criteria to be included in data summary tables? Yes No

44. Identify the criteria that must be presented in the summary tables.

_____
_____
_____

45. Will State, Federal, or Regional criteria be used to select COPCs? Yes No

46. Identify the criteria to be used as COPC selection tools.

_____
_____
_____

47. Are filtered and unfiltered surface water samples differentiated? Yes No

48. If such samples are differentiated, how?

_____
_____
_____

49. Which of these samples will be used for the human health risk assessment?

Surface Water Filtered Unfiltered
Groundwater Filtered Unfiltered

50. Which of these samples will be used for the ecological assessment?

Surface Water	Filtered	Unfiltered
Groundwater	Filtered	Unfiltered

51. Will background data be included in the database? Yes No

52. How are background samples identified?

_____

_____

_____

53. Will background results be used to support selection of COPCs? Yes No

53. What statistical analyses will be required for the background data?

_____

_____

_____

54. Will background data be segregated by depth? Yes No

55. What background matrices must be segregated by depth?

_____

_____

_____

56. What format will be used for data presentation (e.g., appendices and summary tables, comprehensive text tables, tag maps, isoconcentration contours, etc.)?

_____

_____

_____

ATTACHMENT 4

SAMPLE TRACKING REQUEST FORM

Sample Tracking and Data Management at Project Inception

PROJECT START-UP CHECKLIST

ATTACHED IS A PROJECT START-UP CHECKLIST (CAN BE FOUND IN DATA MANAGEMENT ROOM). WHENEVER A NEW PROJECT IS STARTED THE TOP PART SHOULD BE FILLED IN. A COPY SHOULD BE RETURNED TO THE DATA MANAGEMENT ROOM. KEEP ORIGINAL FOR YOUR RECORDS TO KEEP TRACK OF WHAT HAS BEEN PROVIDED. IMSG WILL CHECK OFF WHEN ALL INFORMATION IS RECEIVED

FOLLOWING THIS PROCESS WILL IMPROVE THE FOLLOWING:

- TURN-AROUND TIME FOR DELIVERABLES NEEDED WHEN ALL RESULTS HAVE BEEN RECEIVED.
- CONFIDENCE THAT ALL SAMPLE RESULTS HAVE BEEN RECEIVED
- CONSISTENCY OF SAMPLE NOMENCLATURE
- CORRECTNESS OF SAMPLE ATTRIBUTES
- REVIEW OF INVOICES
- ENABLE IMSG PERSONNEL TO BETTER TRACK UPCOMING WORKLOAD

PROJECT START-UP CHECKLIST
INFORMATION NEEDED TO CREATE NEW DATABASE

PROJECT NAME:_____

CTO #:_____ JOB #:_____

PROJECT MANAGER/CONTACT:_____

LABELS: Y / N DUE DATE:_____

VALIDATE: Y / N / L DUE DATE:_____

COMBINE WITH HISTORICAL DATA: Y/N

SAMPLE DATA CHECKLIST:

_____ SAMPLE NUMBERS AND ANALYSES (LOCATIONS,DEPTHS)
_____ SECTION OF WORKPLAN PERTAINING TO SAMPLE NOMENCLATURE
_____ LABORATORY/BOTTLE REQUIREMENTS
_____ LAB SPECS
_____ COC'S
_____ SAMPLE LOG SHEETS
_____ DUPLICATE ID'S / ORIGINALS
_____ SURVEY DATA / SAMPLE LOCATION MAPS
_____ BREAKDOWN OF PROJECT BY SITE / MATRIX FOR FUTURE PRINTOUTS
_____ TABLE HEADERS (SEE EXAMPLE)

TO BE COMPLETED BY IMSG:

_____ FINAL RESULTS GIVEN TO _____ (PM/IMSG)
DATE:_____
_____ SAMPLE DATA LOADED INTO NEW/EXISTING PROJECT DATABASE
_____ RESULTS LOADED INTO NEW/EXISTING PROJECT DATABASE
PATHNAME OF PROJECT DATABASE:_____
_____ DATA LOADED INTO GIS

ATTACHMENT 5

SAMPLE TRACKING DATABASE EXAMPLE

CTO 020 SDG U06972

07/15/98

Proj Name	Job No	Sdg	Sample Number	Lab Id	Fraction	Sort	Lab Rec	B&R Rec	Turn-Time	WO No	Laboratory
PARRIS ISLAND	7394	U06972	PAI-03-SW-010	9806G972-001	LV	LV	06/03/98	07/14/98	41	9806G972	RECRA
PARRIS ISLAND	7394	U06972	PAI-03-SW-010	9806G972-001	MISC	CN	06/03/98	07/14/98	41	9806G972	RECRA
PARRIS ISLAND	7394	U06972	PAI-03-SW-010	9806G972-001	MISC	CR6	06/03/98	07/14/98	41	9806G972	RECRA
PARRIS ISLAND	7394	U06972	PAI-03-SW-010	9806G972-001	OS	OS	06/03/98	07/14/98	41	9806G972	RECRA
PARRIS ISLAND	7394	U06972	PAI-03-SW-010	9806G972-001	PAH	PAH	06/03/98	07/14/98	41	9806G972	RECRA
PARRIS ISLAND	7394	U06972	PAI-03-SW-010	9806G972-001	PEST/PCB	PCB	06/03/98	07/14/98	41	9806G972	RECRA
PARRIS ISLAND	7394	U06972	PAI-03-SW-010	9806G972-001	PEST/PCB	PEST	06/03/98	07/14/98	41	9806G972	RECRA

ATTACHMENT 6

EXAMPLE SAMPLE JAR LABELS

Tetra Tech NUS, Inc.
661 Andersen Drive
Pittsburgh, 15220
(412)921-7090

Project: NWS CHARLESTON
Location: 14SB01

Sample No: 14-P-001-01 | Matrix: SOIL
Date: | Time: | Preserve: 4° C
Analysis: TCL Volatiles
Sampled by: | Laboratory: RECRA

Tetra Tech NUS, Inc.
661 Andersen Drive
Pittsburgh, 15220
(412)921-7090

Project: NWS CHARLESTON
Location:

Sample No: | Matrix:
Date: | Time: | Preserve:
Analysis:
Sampled by: | Laboratory:

Tetra Tech NUS, Inc.
661 Andersen Drive
Pittsburgh, 15220
(412)921-7090

Project: NWS CHARLESTON
Location: 14SB01

Sample No: 14-P-001-01 | Matrix: SOIL
Date: | Time: | Preserve: 4° C
Analysis: TCL Semivolatiles, TAL Metals, Cyanide
Sampled by: | Laboratory: RECRA

Tetra Tech NUS, Inc.
661 Andersen Drive
Pittsburgh, 15220
(412)921-7090

Project: NWS CHARLESTON
Location:

Sample No: | Matrix:
Date: | Time: | Preserve:
Analysis:
Sampled by: | Laboratory:

Tetra Tech NUS, Inc.
661 Andersen Drive
Pittsburgh, 15220
(412)921-7090

Project: NWS CHARLESTON
Location: 14SB01

Sample No: 14-P-001-01 | Matrix: SOIL
Date: | Time: | Preserve: 4° C
Analysis: OTTO Fuel
Sampled by: | Laboratory: GEL

Tetra Tech NUS, Inc.
661 Andersen Drive
Pittsburgh, 15220
(412)921-7090

Project: NWS CHARLESTON
Location:

Sample No: | Matrix:
Date: | Time: | Preserve:
Analysis:
Sampled by: | Laboratory:

Tetra Tech NUS, Inc.
661 Andersen Drive
Pittsburgh, 15220
(412)921-7090

Project: NWS CHARLESTON
Location:

Sample No: | Matrix:
Date: | Time: | Preserve:
Analysis:
Sampled by: | Laboratory:

Tetra Tech NUS, Inc.
661 Andersen Drive
Pittsburgh, 15220
(412)921-7090

Project: NWS CHARLESTON
Location:

Sample No: | Matrix:
Date: | Time: | Preserve:
Analysis:
Sampled by: | Laboratory:

Tetra Tech NUS, Inc.
661 Andersen Drive
Pittsburgh, 15220
(412)921-7090

Project: NWS CHARLESTON
Location:

Sample No: | Matrix:
Date: | Time: | Preserve:
Analysis:
Sampled by: | Laboratory:

Tetra Tech NUS, Inc.
661 Andersen Drive
Pittsburgh, 15220
(412)921-7090

Project: NWS CHARLESTON
Location:

Sample No: | Matrix:
Date: | Time: | Preserve:
Analysis:
Sampled by: | Laboratory:

ATTACHMENT 7

ELECTRONIC DATA DELIVERABLE REQUIREMENTS FOR ANALYTICAL LABORATORIES

ELECTRONIC DATA FORMAT REQUIREMENTS

1.0 INTRODUCTION

The laboratory is to provide 3.5" high density diskette(s) containing separate database (DBF) files in the format specified in this Attachment. The electronic deliverable includes all environmental samples. sample dilutions, sample reanalyses, and laboratory quality control samples. **All entries in the electronic deliverable must agree exactly with the final entries reported on the hardcopy data package sample result summaries.** Any corrections made to the hardcopy data must also be made to the electronic file. Appropriate qualifiers as identified by the analytical protocol must also be designated; laboratory QC non-compliance codes are not to be depicted.

Each diskette is to be properly labeled with the laboratory name, project name, file name(s), and laboratory point of contact. Electronic files should be delivered in the same fashion as are the hard copy data packages. A separate .dbf file shall be made for each analytical fraction (by method) and each sample delivery group (SDG). The files shall be named with the first character being the analytical fraction designator, followed by an underscore, followed by the SDG name. For example, the file for the volatile fraction for SDG BR001 should be named V_BR001.DBF. Additionally, the laboratory must provide a hardcopy listing all electronic files saved to the diskette, indicating what analytical fraction and matrix the file data contained therein pertain to. All electronic data deliverables are due within the same time established for the associated hardcopy data packages.

In addition, the laboratory QC officer must read and sign a copy of the Quality Assurance Re Form displayed on the next page of this Attachment. Electronic deliverables are not considered to be complete without the accompanying Quality Assurance Review Form.

I _____, as the designated Quality Assurance Officer, hereby attest that all electronic deliverables have been thoroughly reviewed and are in agreement with the associated hardcopy data. The enclosed electronic files have been reviewed for accuracy (including significant figures), completeness and format. The laboratory will be responsible for any labor time necessary to correct enclosed electronic deliverables that have been found to be in error. I can be reached at (_____)_____if there are any questions or problems with the enclosed electronic deliverables.

Signature:_____ Title: _____ Date: _____

Revision 4
KAC 11-11-97

The analytical data shall be delivered electronically in a Dbase III file format (filename.dbf). The exact structure of the database is described in the table below. It shall be the responsibility of the laboratory to ensure that all electronic entries are in strict accordance with the information provided on the Form I.

An example database shall be sent for review prior to the first electronic deliverable in Dbase III format. The example file will be examined for completeness and comments will be sent to the laboratory. Any questions regarding the electronic deliverable shall be directed to Patrick Hooper at Brown and Root Environmental (412)921-8250.

DATA FIELD	DATA TYPE	FIELD WIDTH	DATA FIELD DESCRIPTION
SAMPLE_NO	C	25	Field sample ID as listed on the chain-of-custody. The sample number indicated in this field should never be truncated. The only exception for this field not matching the chain-of-custody is for reanalyses and matrix spike results in which a RE or MS suffix will be added to the sample number respectively.
TRUNCATE	C	15	If the field sample ID listed on the Chain of Custody is truncated by the laboratory for use with the laboratory software, the truncated sample ID should appear in this field.
LAB_ID	C	15	Laboratory number for the given sample.
LABORATORY	C	25	Laboratory name.
BATCH_NO	C	10	Laboratory code for batch of samples included in a given run.
ASSOC_BLNK	C	15	Laboratory name of the method blank associated with that particular batch of samples.
QC_TYPE	C	15	Normal Environmental Sample = "NORMAL", Laboratory Duplicate = "DUPLICATE", Matrix Spike = "MS", Matrix Spike Duplicate = "MSD", Laboratory Control Sample = "LCS", Laboratory Control Sample Duplicate = "LCSD", Method Blank = "M_BLANK", Preparation Blank = "P_BLANK".
SAMP_DATE	D	8	Date of sample collection as indicated on the Chain of Custody. Example: 11/07/93.
REC_DATE	D	8	Date sample was received by the laboratory.
EXTR_DATE	D	8	Date sample was extracted or prepared by the laboratory.
ANAL_DATE	D	8	Date sample was analyzed by the laboratory.
RUN_NUMBER	N	2 (0)	The number of the analytical run for a given sample in sequence. For example, if a sample is diluted and reanalyzed, the original run number would be 1 and the reanalysis would be 2.
SDG	C	15	Sample delivery group identifier assigned by the laboratory. This number should exactly match the SDG designated on the hardcopy data package.

DATA FIELD	DATA TYPE	FIELD WIDTH	DATA FIELD DESCRIPTION
PROJECT_NO	C	10	Identification of Project Number or CLEAN Task Order (CTO) number.
PROJ_MNGR	C	25	The Brown & Root Project Manager's last name, followed by a comma, followed by the first initial of the Project Manager (e.g. Hutson, D).
PARAMETER	C	45	Chemical or analyte name exactly as reported on Form I.
CAS_NO	C	10	Chemical Abstract Service number for the parameter listed. The CAS number should be reported exactly as it is listed in publications such as the Merck Index. This field should be left blank for those parameters not having CAS numbers (e.g. Total Organic Carbon).
FRACTION	C	5	Metals = 'M', Volatiles = 'OV', Semivolatiles/BNAs = 'OS', Pesticides = 'PEST', Herbicides = 'HERB', Polychlorinated Biphenyls = 'PCB', Explosives = 'EXP', Any petroleum hydrocarbon or fuel = 'TPH', Wet Chemistry = 'WET', Radionuclide = 'RAD', Miscellaneous = 'MISC'
METHOD	C	20	Analytical method used to quantitate parameter concentrations as listed in the laboratory technical specification (e.g. '8270A' for SW-846 Method 8270A.
LAB_RESULT	N	20 (6)	Reported value in units specified in the UNITS field containing the proper number of significant digits. The % Recovery shall be placed in this field for matrix spike and laboratory control sample results.
UNITS	C	5	The units of measure as reported on the Form I.
LAB_QUAL	C	2	The laboratory qualifier as reported on the Form I. For example, a 'U' qualifier should be used for all nondetected results.
IDL	N	15 (6)	Instrument detection limit in units specified in the UNITS field.
MDL	N	15 (6)	Method detection limit in units specified in the UNITS field and method specified in the METHOD field.
CRDL_CRQL	N	15 (6)	Contract Required Detection/Quantitation Limit in the units specified in the UNITS field. RDL for non-CLP parameters.
DIL_FACTOR	N	6 (1)	Dilution factor.
PCT_MOIST	N	5 (1)	Percent moisture for soil samples; blank for water samples.
COMMENTS	C	20	Analytical result qualifier or comment other than that listed in the LAB_QUAL field. Example: 'Reanalysis'.

C = Character string (everything shall be reported in capital letters)
N = Numeric string (decimal places are in parentheses in field width column)
D = Date (Ex: 05/25/97)

ATTACHMENT 8

DATABASE STRUCTURE

Database Structure

The NAS Dallas master database shall contain 18 standard tables to store all chemical, geological, and hydrogeological data. The structure, indexes, primary keys, and relations for each table are defined below.

TABLE: well
PRIMARY KEY: location

Table Structure

FIELD	TYPE	DESCRIPTION
Location	C (25)	Unique location name.
post_id	C (20)	Location name as derived from original source document.
instal_date	D (8)	Date the monitoring well was installed. Null for other location types.
loc_type	C (4)	Type of location.
Northing	N (15,4)	Northing coordinate in horizontal datum referenced in the HORIZ_DATUM field.
Easting	N (15,4)	Easting coordinate in horizontal datum referenced in the HORIZ_DATUM field.
horiz_datum	C (8)	Datum in which the horizontal coordinates were derived.
grnd_surf	N (15,4)	Ground surface elevation with reference to mean sea level in vertical datum referenced in the VERT_DATUM field
vert_datum	C (25)	Datum in which the vertical coordinates were derived.
datum_state	C (2)	State for which datum was developed
Surveyed	L (1)	Logical field denoting whether positional data were surveyed or digitized.
Surveyor	C (50)	Company who performed the survey.
survey_date	D (8)	Date in which survey was performed.
surv_method	C (25)	Surveying method used.
Longitude	N (15,4)	Longitude
Latitude	N (15,4)	Latitude

Table Indexes

INDEX	TYPE
Location	Primary
Hd	Regular
Vd	Regular
loc_type	Regular

Table Relations:

Relation 1
*RelatedChild loc_type
*RelatedTable loc_type_vvl
*RelatedTag loc_type

Relation 2
*RelatedChild hd
*RelatedTable horiz_datum_vvl
*RelatedTag hd

Relation 3
*RelatedChild vd
*RelatedTable vert_datum_vvl
*RelatedTag vd

TABLE: loc_type_vvl - Valid value list for LOC_TYPE field in the well table.
PRIMARY KEY: loc_type

Table Structure

FIELD	TYPE	DESCRIPTION
loc_type	C (4)	Location type
Description	C (40)	Description of location type

Table Indexes

INDEX	TYPE
loc_type	Primary

TABLE: horiz_datum_vvl - Valid value list for HORIZ_DATUM field in the well table.
PRIMARY KEY: hd (horiz_datum)

Table Structure

FIELD	TYPE	DESCRIPTION
Horiz_datum	C (15)	Datum in which x,y coordinates reflect

Table Indexes

INDEX	TYPE
hd (horiz_datum)	Primary

Table Indexes

INDEX	TYPE
loc_type	Primary

TABLE: vert_datum_vvl - Valid value list for VERT_DATUM field in the well table.
PRIMARY KEY: vd (vert_datum)

Table Structure

FIELD	TYPE	DESCRIPTION
Vert_datum	C (15)	Datum in which z coordinate reflects

Table Indexes

INDEX	TYPE
vd (vert_datum)	Primary

TABLE: sample_data - Sample data table
PRIMARY KEY: nsample

Table Structure

FIELD	TYPE	DESCRIPTION
Location	C (25)	Unique location name.
Matrix	C (4)	Sample matrix
Nsample	C (35)	Unique sample identification
Sample	C (25)	Sample identification as designated on Chain-of-Custody
Sacode	C (8)	Sample code for reference to field duplicates
top_depth	N (5,1)	Depth in feet to the top of the sample interval. Applicable for soil and sediment samples.
Bottom_depth	N (5,1)	Depth in feet to the bottom of the sample interval. Applicable for soil and sediment samples. *Rule Expression*: if(bottom_depth>0.top_depth<=bottom_depth)
qc_type	C (2)	Quality control type

Status	C (10)	Status of sample location – Normal or excavated
sample_date	D (8)	Date in which sample was collected
Validated	L (1)	Logical field denoting whether or not data validation was performed on sample
coll_method	C (10)	Sample collection method
cto_proj	C (5)	Clean task order (Navy) or project number in which the sample was collected (e.g. "129")
proj_manager	C (25)	Internal projcect manager for which the data was originally generated (e.g. "Hutson, D.").

Table Indexes

INDEX	TYPE
Location	Regular
Nsample	Primary
Sacode	Regular
Matrix	Regular
Status	Regular
qc_type	Regular
coll_meth	Regular

Table Relations:

Relation 1
*RelatedChild sacode
*RelatedTable sacode_vvl
*RelatedTag sacode

Relation 2
*RelatedChild qc_type
*RelatedTable qc_type_vvl
*RelatedTag qc_type

Relation 3
*RelatedChild matrix
*RelatedTable matrix_vvl
*RelatedTag matrix

Relation 4
*RelatedChild location
*RelatedTable well
*RelatedTag location

Relation 5
*RelatedChild coll_meth
*RelatedTable coll_method_vvl
*RelatedTag coll_meth

TABLE: sacode_vvl - Sample code valid value list for SACODE field in sample_data.dbf
PRIMARY KEY: sacode

Table Structure

FIELD	TYPE	DESCRIPTION
Sacode	C (8)	Sample code designating whether sample is a normal environmental sample, a field duplicate or the average of field duplicate pairs
Description	C (30)	Description of sacode entry

Table Indexes

INDEX	TYPE
Sacode	Primary

TABLE: qc_type_vvl - Quality control valid value list for QC_TYPE field in sample_data.dbf
PRIMARY KEY: qc_type

Table Structure

FIELD	TYPE	DESCRIPTION
qc_type	C (10)	Quality control type
Description	C (30)	Description of quality control type

Table Indexes

INDEX	TYPE
qc_type	Primary

TABLE: matrix_vvl - Matrix valid value list for MATRIX field in sample_data.dbf
PRIMARY KEY: matrix

Table Structure

FIELD	TYPE	DESCRIPTION
Matrix	C (4)	Sample matrix
Description	C (25)	Description of sample matrix code

Table Indexes

INDEX	TYPE
Matrix	Primary

TABLE: well_completion
PRIMARY KEY: None

Table Structure

FIELD	TYPE	DESCRIPTION
Location	C (25)	Unique location name
top_casing	N (8,2)	Elevation of top of well casing in vertical datum found in VERT_DATUM in the well table
hole_diameter	N (5,1)	Diameter of the drilled hole in inches *Rule Expression*: hole_diameter>casing_id.AND.hole_diameter>casing_od
scr_aquifer	C (30)	Aquifer name in which the screen resides
screen_material	C (15)	Type of material in which the screen is constructed from
scrn_slot_size	N (5,1)	Screen slot size in thousandths of an inch
scrn_top_depth	N (5,2)	Depth below ground surface to the top of the screen (in feet)
scrn_bot_depth	N(5,2)	Depth below ground surface to the bottom of the screen *Rule Expression*: if(scrn_bot_depth>0,scrn_top_depth<scrn_bot_depth)
scrn_top_elev	N (6,2)	Elevation the top of the screen in vertical datum found in VERT_DATUM in the well table.
scrn_bot_elev	N (6,2)	Elevation the top of the screen in vertical datum found in VERT_DATUM in the well table. *Rule Expression*: if(scrn_bot_elev>0.scrn_top_depth>scrn_bot_depth)
drill_method	C (15)	Drilling method for well installation
Contractor	C (20)	Drilling contractor
casing_material	C (15)	Type of material in which the casing is constructed from
depth_to_seal	N (8,2)	Depth below ground surface to seal (in feet)

seal_material	C (15)	Type of material in which the seal is constructed from
fill_top_depth	N (6,2)	Depth below ground surface to the top of fill material (in feet)
fill_bot_depth	N (6,2)	Depth below ground surface to the bottom of fill material (in feet). *Rule Expression*: if(fill_bot_depth>0,fill_top_depth<scrn_bot_depth)
fill_material	C (20)	Type of material used for fill.
comments	M (4)	Geologist's comments

Table Indexes

INDEX	TYPE
location	Regular

Table Relations:
Relation 1
*RelatedChild location
*RelatedTable well
*RelatedTag location

TABLE: lithology
PRIMARY KEY: None

FIELD	TYPE	DESCRIPTION
location	C (25)	Unique location name
top_lithology	N (6,2)	Depth in feet below ground surface to the top of lithologic unit
bottom_lithology	N (6,2)	Depth in feet below ground surface to the bottom of lithologic unit
uscs_code	C (5)	Unified Soil Classification Service Code for lithology type
blow_counts	C (8)	Number of blow counts recorded on boring log
description	C (80)	Geologist's description of lithology
comments	M (4)	Geologist's comments.

Table Indexes

INDEX	TYPE
location	Regular
uscs_code	Regular

Table Relations:
Relation 1
*RelatedChild location
*RelatedTable well
*RelatedTag location
Relation 2
*RelatedChild uscs_code
*RelatedTable lithology_vvl
*RelatedTag uscs_code

TABLE: lithology_vvl - Lithology valid value list for USCS_CODE field in lithology.dbf
PRIMARY KEY: uscs_code

Table Structure

FIELD	TYPE	DESCRIPTION
uscs_code	C (4)	Unified Soil Classification Service Code for lithology type
descript	C (70)	Description of lithology for given USCS code

Table Indexes

INDEX	TYPE
uscs_code	Primary

TABLE: coll_method_vvl - Collection method valid value list for COLL_METHOD field in sample_data.dbf
PRIMARY KEY: coll_meth

Table Structure

FIELD	TYPE	DESCRIPTION
coll_method	C (10)	Sample collection method

Table Indexes

INDEX	TYPE
coll_method	Primary

TABLE: cas_vvl - CAS number valid value list for CAS field in analytical results.dbf
PRIMARY KEY: cas

Table Structure

FIELD	TYPE	DESCRIPTION
parameter	C (40)	Parameter or chemical name
cas	C (11)	Chemical Abstracts Service Number

Table Indexes

INDEX	TYPE
parameter	Regular
cas	Primary

TABLE: analytic_results
PRIMARY KEY: nfp (nsample+fraction+parameter)

Table Structure

FIELD	TYPE	DESCRIPTION
nsample	C (35)	Unique sample identification
lab_id	C (15)	Laboratory sample identification
laboratory	C (25)	Laboratory name
batch_no	C (10)	Analytical batch number
assoc_blnk	C (15)	Associated blank
extr_date	D (8)	Extraction date
anal_date	D (8)	Analysis date
run_number	I (4)	Sequential analytical run number
sdg	C (15)	Sample delivery group
parameter	C (45)	Parameter or chemical name (using IUPAC nomenclature where appropriate)
cas	C (11)	Chemical Abstracts Service Number
fraction	C (5)	Analtytical fraction
method	C (20)	Analytical method
lab_result	N (20,6)	Analytical result as reported by the laboratory
lab_qual	C (5)	Qualifier as reported by the laboratory
val_res	N (20,6)	Final result (via validation or otherwise)

result	C (20)	Final analytical result with the correct number of significant figures
val_qual	C (3)	Validation qualifer (null if data were not validated)
qual	C (3)	Final qualifer (validation or otherwise)
units	C (5)	Units of measure for the RESULT field
idl	N (15,6)	Instrument detection limit (same units as UNITS field)
mdl	N (15,6)	Method detection limit (same units as UNITS field)
crdl_crql	N (15,6)	Contract required detection/quantitation limit (same units as UNITS field)
dil_factor	N (6,1)	Dilution factor
pct_moist	N (5,1)	Percent moisture
comments	C (20)	Comments from laboratory analyst

Table Indexes

INDEX	TYPE
nfp	Primary
units	Regular
qual	Regular
fraction	Regular
parameter	Regular
nsample	Regular
cas	Regular

Table Relations:

Relation 1
*RelatedChild cas
*RelatedTable cas_vvl
*RelatedTag cas
Relation 2
*RelatedChild units
*RelatedTable units_vvl
*RelatedTag units
Relation 3
*RelatedChild qual
*RelatedTable qual_vvl
*RelatedTag qual
Relation 4
*RelatedChild fraction
*RelatedTable fraction_vvl
*RelatedTag fraction
Relation 5
*RelatedChild parameter
*RelatedTable para_vvl
*RelatedTag para
Relation 6
*RelatedChild nsample
*RelatedTable sample_data
*RelatedTag nsample

TABLE: units_vvl - Units valid value list for UNITS field in analytical_results.dbf
PRIMARY KEY: Units

Table Structure

FIELD	TYPE	DESCRIPTION
Units	C (8)	Units of measure for chemical analysis
Description	C (20)	Description of units

Table Indexes

INDEX	TYPE
Units	Primary

TABLE: qual_vvl
PRIMARY KEY: qual - Qualifier valid value list for QUAL field in analtyic_results.dbf

Table Structure

FIELD	TYPE	DESCRIPTION
Qual	C (5)	Final QA qualifier
Description	C (60)	Definition of qualifier

Table Indexes

INDEX	TYPE
Qual	Primary

TABLE: fraction_vvl - Analytical fraction valid value list for FRACTION field in analytic_results.dbf
PRIMARY KEY: fraction

Table Structure

FIELD	TYPE	DESCRIPTION
Fraction	C (10)	Analytical fraction
Description	C (35)	Description of fraction

Table Indexes

INDEX	TYPE
Fraction	Primary

TABLE: para_vvl
PRIMARY KEY: parameter

Table Structure

FIELD	TYPE	DESCRIPTION
Para	C (60)	Parameter or chemical name
frac_name	C (35)	Analytical fraction for given parameter

Table Indexes

INDEX	TYPE
Para	Primary

TABLE: fluid
PRIMARY KEY: None

Table Structure

FIELD	TYPE	DESCRIPTION
Location	C (25)	Unique location name
Fluid_date	D (8)	Date measurement was taken
meas_elev	N (8,2)	Measuring point elevation
dep_to_water	N (6,2)	Depth below ground surface to water table (in feet)
dep_to_fp	N (6,2)	Depth below ground surface to free product (in feet)
elev_water	N (8,2)	Elevation of water level
elev_fp	N (8,2)	Elevation of free product
prod_thick	N (6,2)	Product thickness in feet

Table Indexes

INDEX	TYPE
location	Regular

Table Relations:
Relation 1
*RelatedChild location
*RelatedTable well
*RelatedTag location

ATTACHMENT 9

ARCVIEW GIS STRUCTURE

ArcView GIS Structure

The NAS Dallas ArcView GIS shall have the following directory structure and database table structure.

Part One: Directory Structure

The following table defines the directory structure and major file names/types located within each directory.

Main subdirectory	First tier subdirectories	Second tier subdirectories	Files/Types
p:\gis\project name\	database\		coordinate.dbf cross_reference.dbf res_ gw.dbf res_so.dbf res_sd.dbf res_sw.dbf well_completion.dbf
		criteria\	crit_ gw.dbf crit_so.dbf crit_ sd.dbf crit_sw.dbf crit_des.dbf
	mapping\	aerial\	registered aerial photos
		drg\	USGS Digital Raster Graphic
		image\	GeoStatistic Layers, pictures of sites, equipment, EVS, and all other raster files.
		dwg\	AutoCAD files
		dgn\	Microstation files
		shp\	samp_gw.shp .dbf .shx samp_so.shp .dbf .shx samp_sd.shp .dbf .shx samp_sw.shp .dbf .shx and all other AV shape files
	working\	database\	files used to generate specific drawings will be put under the working subdirectory in subdirectories similar to database & mapping. These will not be included in CD deliverable.
		mapping\	same as above

Part Two: Database Table Structure

The ArcView GIS will contain separate database tables to store analytical, criteria, and coordinate information. The structure of these tables is presented below.

Analyticl Data Table

The following table lists all the fields contained in the analytic database table.

FIELD	VISIBLE	ALIAS	DESCRIPTION
site	Yes	Site or SWMU	Site or SWMU
location	Yes	Location	Unique location name
nsample	Yes	Sample	Unique sample identification
sample	No		Sample identification as designated on Chain-of-Custody
sample_date	Yes	Sample Date	Date in which sample was collected
matrix	Yes	Matrix	Sample matrix
sacode	Yes	Sample Code	Sample code for reference to field duplicates
depth	Yes	Depth	Depth in feet to the middle of the sample interval. Applicable for soil and sediment samples.
top_depth	Yes	Top Depth	Depth in feet to the top of the sample interval. Applicable for soil and sediment samples.
bottom_depth	Yes	Bottom Depth	Depth in feet to the bottom of the sample interval. Applicable for soil and sediment samples. Rule Expression: if(bottom_depth>0,top_depth<=bottom_depth)
parameter	Yes	Parameter	Parameter or chemical name (using IUPAC nomenclature where appropriate)
cas	Yes	CAS	Chemical Abstracts Service Number
fraction	Yes	Fraction	Analytical fraction
val_res	Yes	Numeric Result	Final result (via validation or otherwise)
qual	Yes	Qualifier	Final qualifier (validation or otherwise)
units	Yes	Units	Units of measure for the RESULT field
method	Yes	Method	Analytical method
status	Yes	Status	Status of sample location – Normal or excavated
validated	Yes	Validated	Logical field denoting whether or not data validation was performed on sample
coll_method	Yes	Collection Method	Sample collection method
cto_proj	Yes	CTO	Clean task order (Navy) or project number in which the sample was collected (e.g. "129")
proj_manager	Yes	Project Manager	Internal project manager for which the data was originally generated (e.g. "Hooper, P.").
lab_id	No	Laboratory ID	Laboratory sample identification
laboratory	No	Laboratory	Laboratory name
batch_no	No	Batch Number	Analytical batch number

assoc_blnk	No	Associated Blank	Associated blank
extr_date	No	Extraction Date	Extraction date
anal_date	No	Analysis date	Analysis date
run_number	No	Run Number	Sequential analytical run number
sdg	No	SDG	Sample delivery group
lab_result	No	Result	Analytical result as reported by the laboratory
lab_qual	No	Lab Qualifier	Qualifier as reported by the laboratory
result	No	String Result	Final analytical result with the correct number of significant figures
val_qual	No	Validation Qualifier	Validation qualifier (null if data were not validated)
idl	No	Detection Limit	Instrument detection limit (same units as UNITS field)
mdl	No	Detection Units	Method detection limit (same units as UNITS field)
crdl_crql	No		Contract required detection/quantitation limit (same units as UNITS field)
dil_factor	No	Dilution factor	Dilution factor
pct_moist	No	Percent moisture	Percent moisture
ourresult	No		
qc_type	No		Quality control type
comments	No	Comments	Comments from laboratory analyst

Criteria Table

Each medium will have a criteria table to specify the applicable criteria for all parameters.

FIELD	ALIAS	DESCRIPTION
parameter	Parameter	Parameter or chemical name (using IUPAC nomenclature where appropriate)
epa_mcl	None	Federal MCL – groundwater

Note: usually there will be many criteria fields. This example table only shows the "epa_mcl" criteria field.

Criteria Description Table

This table stores the definition or description of all standards and criteria used in the project. For example, epa_mcl's media would be GW, description would be "Federal Maximum Contaminant Level".

FIELD	Visible	DESCRIPTION
Field	Yes	
Media	Yes	
Descript	Yes	

Coordinate Table

The coordinate table holds all the geographic position information of sampling locations

FIELD	Visible	ALIAS	DESCRIPTION
Location	Yes	None	Unique location name.
post_id	Yes	Location Designation	Location name as derived from original source document.
instal_date	No	Installation Date	Date the monitoring well was installed. Null for other location types.
loc_type	Yes	Location Type	Type of location. Example MW, HP, etc.
northing	Yes		Northing coordinate in horizontal datum referenced in the HORIZ_DATUM field.
Easting	Yes		Easting coordinate in horizontal datum referenced in the HORIZ_DATUM field.
Grnd_surf	Yes	Ground Surface Elevation	Ground surface elevation with reference to mean sea level in vertical datum referenced in the VERT_DATUM field
horiz_datum	Yes	Horizontal Datum	Datum in which the horizontal coordinates were derived.
Vert_datum	Yes	Vertical Datum	Datum in which the vertical coordinates were derived.
Datum_state	Yes	Coordinate System	State for which datum was developed
Surveyed	Yes		Logical field denoting whether positional data were surveyed or digitized.
Surveyor	Yes		Company who performed the survey.
Survey_date	No	Survey Date	Date in which survey was performed.
Surv_method	No	Survey Method	Surveying method used.
Longitude	No		Longitude
Latitude	No		Latitude
gw_code	Yes		This will be populated by database personnel. It will be used for event driven theme.
Sd_code	Yes		This will be populated by database personnel. It will be used for event driven theme.
So_code	Yes		This will be populated by database personnel. It will be used for event driven theme.
Sw_code	Yes		This will be populated by database personnel. It will be used for event driven theme.
_nullflags	No		Various fields are put in by database starting here and followed by several fields. Make all of these invisible

APPENDIX D

FIELD AUDIT CHECKLIST
(EXAMPLE)

Location: NSWC Crane, Crane, Indiana

Project: NSWC Crane Soils Background Study

Date of Audit: _____

Instructions: Record answers to questions below, providing comments as required to clarify the answers.

QA/QC Procedures

1. Were any field observations, deficiencies, non-conformances, 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, or WP?

   _____

6. For all sites, were field duplicates submitted “blind” to the 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, or WP?

_____
_____
_____

Soil Sampling

8. Are the sampling devices designated in the WP, or applicable TtNUS SOP being used?

_____
_____
_____
_____

9. Was the following information recorded in the boring logs or the field notebook?

For soil classification:

Was the USCS classification and soil type (clay, silt, sand) indicated?

_____

Were the following characteristics indicated per the relevant TtNUS SOP CTO 83-4 sections?

color _____
soil type _____
relative density and consistency _____
weight percentage _____
moisture _____
stratification _____
texture/fabric/bedding _____

10. 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 pin flag _____
sketch approximate locations of sample points in site notebook _____

Equipment Decontamination Procedures

11. Has an adequate pre-determined area for steam cleaning of equipment been established?

_____
_____

12. Is the decontamination (decon) area lined and/or bermed?

_____
_____

13. Are augers decontaminated by steam cleaning as required?

_____
_____

14. Was steam cleaning conducted:

prior to commencement of field activities? _____
between boring/pit locations? _____
at the end of field activities?_____

15. Verify that all sampling equipment not subject to steam cleaning (e.g., trowels, mixing bowls, etc.) are subjected to decontamination per the sequence outlined in the project planning documents.

_____
_____

Waste Handling Procedures

16. Were cuttings or fluids disposed of in accordance with project planning documents (i.e., discharged to ground, drummed, or tanked)?

_____

17. Do the project planning documents provide for the disposal of Personal Protective Equipment (PPE) by double-bagging and discard?

_____

18. By what method are PPE disposed of?

_____

19. If applicable, were used spill-containment materials containerized or otherwise acceptably disposed of?

_____
_____

Sample Handling

20. Are the appropriate containers provided by the laboratory being used for each sample?

_____
_____
_____

21. Has the temperature blank been handled properly and one submitted with each cooler of samples?

_____
_____

22. Have equipment rinsate blanks of the proper type and frequency been obtained?

_____
_____

23. Have source water blanks been obtained from water sources applicable to the field effort?

_____
_____

24. Have the rinsate and field blanks been designated for the same analyses as the associated samples?

_____
_____

25. 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 possession
- it 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

26. Are all sample logs complete (i.e., containing all information stipulated in SOP CTO 83-4)?

_____
_____

27. Have chain-of-custody (COC) forms been filled out for all samples, including field quality control samples?

_____
_____

28. Have the COC forms been signed by the appropriate individual at each step that the samples are relinquished?

_____
_____

29. Have the COC forms been filled-out using black waterproof ink?

_____
_____

30. 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.)

_____
_____

31. Have the appropriate analyses (per the project planning documents) been properly designated for each sample on the chain-of-custody form?

_____
_____

32. Have all sample labels been filled out appropriately and completely?

_____
_____

33. Have sample tags been properly completed and attached securely to samples?

_____
_____

34. Have all sample labels been filled out using indelible ink?

_____
_____

35. Do the sample identifications agree between the sample log, field notebook, sample label and chain-of-custody form?

_____
_____
_____
_____

36. 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?

_____
_____

37. 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 log sheet nos.
- start/completion of boreholes, trenches, monitoring wells
- health and safety issues

38. Is the site logbook a bound notebook with consecutively numbered pages that cannot be easily removed?

_____
_____

39. As required by SOP CTO 83-5, 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 _____

40. As required by SOP CTO 83-5, has the following information been recorded at the beginning of each day?

date _____
start time _____
weather conditions _____
all field personnel present _____
any visitors present _____

41. Do the site logbook entries summarize the daily activities and refer to other site notebooks or log sheets where applicable?

_____

42. Have all site logbook entries been made in black indelible ink?

_____
_____

43. 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.)

_____
_____

44. Did the individual making the logbook entry sign it?

_____

45. Did the Field Operations Leader sign all logbook pages utilized that day at the end of each day?

_____
_____

Auditor Name:_____

Auditor Signature:_____

Date:_____