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

IN REPLY REFER TO

5090
Ser 095/0106

18 MAY 2000

U.S. Environmental Protection Agency, Region V
Waste, Pesticides, & Toxics Division
Waste Management Branch
Illinois, Indiana, and Michigan Section
ATTN: Mr. Peter Ramanauskas (DW-8J)
77 West Jackson Blvd.
Chicago, IL 60604

Dear Mr. Ramanauskas:

In accordance with Appendix I of 40 CFR 270.42, Modification C.2., Crane Division, Naval Surface Warfare Center (NAVSURFWARCENDIV Crane) requests a Class 1 modification to the Final Open Burning Open Detonation (OB/OD) Hazardous Waste Management Permit dated January 13, 2000. The modification revises Standard Operating Procedures (SOPs) in Volume II of the Final Quality Assurance Project Plan for ground water monitoring at the OB/OD units. The following SOPs are revised: LTL-1001, LTL-1004, LTL-1012, LTL-4002, LTL-7202, LTL-8081, and LTL-8276. Persons on the Facility Mailing List will be notified within 90 days after approval of this modification.

The revised SOPs are provided as enclosure (1). The permit required Certification Statement is provided as enclosure (2).

NAVSURFWARCENDIV Crane point of contact is Mr. Thomas J. Brent, Code 09510, telephone 812-854-6160.

Sincerely,

James M. Hunsicker

James M. Hunsicker
Director, Environmental
Protection Department
By direction of the Commander

Encl:
(1) SOP Changes
(2) Certification Statement

Copy to:
ADMINISTRATIVE RECORD
SOUTHNAVFACENGCOM (Code 1829)
IDEM (Doug Griffin)
TTNUS (Ralph Basinski) (w/o encl)
COMARCO (Steve Mehay)
LAUCKS (Hugh Prentice) (w/o encl)

REVISIONS TO
STANDARD OPERATING PROCEDURES
IN
VOLUME II OF THE FINAL QUALITY ASSURANCE PROJECT PLAN
FOR
GROUND WATER MONITORING AT THE OB/OD UNITS.

Enclosure (1)

APPENDIX B

TABLE OF CONTENTS

SOP Number	Revision	Title
Laucks Testing Laboratories, Inc. SOPs:		
• LTL-1001	4	Elements of SOP and Method Formats
• LTL-1002	3	Document Tracking and Control
• LTL-1003	2	Chain-of-Custody and Documentation Procedures
• LTL-1004	4	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	Maintaining Instrument Records and Logbooks
• LTL-1008	5	QC Corrective Action
• LTL-1009	2	Blind Spike Program
• LTL-1011	4	Procedures for the Determination and Reporting of Detection Limits, Reporting Limits, Precision and Accuracy Studies, and Control Limits
• LTL-1012	2	Solvent QC Monitoring for Trace Residue Analysis
• LTL-1013	3	Preparation, Storage, Shelf Life and Traceability Documentation of Standards and Reference Materials
• LTL-1017	1	Internal Audit Procedures
• LTL-1018	0	Overview of Review and Approval Practices for Validatable Data Packages
• LTL-1019	1	Controlling, Maintaining, and Monitoring Laboratory Logbooks
• LTL-1020	1	Integration of IC, GC, HPLC and GCMS Peaks
• LTL-2001	5	Waste Segregation and Disposal
• LTL-3000	11	Extraction Method for Base, Acid, and Neutral Compounds in Water (8270 C by 3520C).
• LTL-3001	1	Extraction Method for Polynuclear Aromatics in Water (8270C by 3520C)
• LTL-3077	3	Extraction Method for Ordnance Compounds in Water (SW 846-8330)
• LTL-3202	1	Extraction Method for Organochlorine Pesticides and Polychlorinated Biphenyls in Water (8081A/ 8082 by 3510C)
• LTL-3510	6	Extraction Method for Chlorinated Herbicides in Water (8151 by 3510C)
• LTL-3550	3	Extraction Method for Picric and Picramic Acids in Water
• LTL-4002	6	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-7009	2	Microwave Assisted Acid Digestion of Aqueous Samples and Extracts Using SW846 Method 3015
• LTL-7010	2	Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FLAA or ICP Spectroscopy by SW846 Method 3010A
• LTL-7101	3	Operation of the Jarrell-Ash Enviro36 Simultaneous ICP
• LTL-7105	0	Method Protocols for the Analysis of Metals by ICP Using SW 846 6010B
• LTL-7202	4	Metals Analysis Using Inductively Coupled Plasma - Mass Spectrometry (ICP/MS) SW846 Method 6020
• LTL-7501	1	Operation of the PE FIMS 400 for the Analysis of Hg
• LTL-8000	1	Determination of Retention Time Windows
• LTL-8019	3	Determination of Non-Halogenated Volatile Organics by Method SW 8015B
• LTL-8081	6	Analysis of Organochlorine Pesticides/PCBs by Method 8081
• LTL-8151	2	Analysis of Chlorinated Herbicides by Method 8151A

APPENDIX B

TABLE OF CONTENTS
PAGE 2 OF 2

SOP Number	Revision	Title
Laucks Testing Laboratories, Inc. SOPs (continued):		
• LTL-8265	1	Determination of Volatile Organic Compounds by Method SW 8260B
• LTL-8276	1	Determination of Semivolatile Organic Compounds by SW-846 Method 8270C
• LTL-8277	0	Determination of Polynuclear Aromatic Hydrocarbon Compounds by Selective Ion Monitoring (SIM) Method 8270C
• LTL-8302	1	HPLC Ordnance 8330 Data Review
• LTL-8303	4	Determination of Picric and Picramic Acids
• LTL-8330	9	Determination of Nitroaromatics and Nitramines by SW-846 Method 8330
• LTL-9104	2	Preparation and Analysis of Total and Amenable Cyanide according to EPA Methods 335.1, 335.3, and SW 846 Method 9012A
• LTL-9108	1	Phosphorus Analysis, All Forms
• LTL-9109	1	Ammonia Analysis by Lachat Auto-Analyzer, EPA Method 350.1
• LTL-9110	6	Determination of Anions by Ion Chromatography by Method 300.0A or 9056
• LTL-9115	2	The Determination of Total Organic Carbon in Water Samples Based on SW846 9060
• LTL-9119	1	Total Organic Halides by SW 846 Method 9020B
• LTL-9125	1	EPA 353.2 Nitrate/Nitrate as N by Lachat
• LTL-9132	1	Determination of Nitrocellulose in Water
• LTL-9205	0	Acid-Soluble Sulfides Distillation Followed by the Titrimetric Procedure for Sulfides SW-846 9030B/9034
Triangle Laboratories, Inc. SOPs:		
• 2.02	1	Quality Assurance Inspections
• 2.04	3	Non-Conformance
• 5.01	5	Sample Receipt, Identification, Handling, and Storage
• CQA125	3	Establishing Method Detection Limits
• DHR182	6	PCDDs and PCDFs by HRGC/HRMS - Method 8290
• DPA104	12	Data Package Assembly and Shipping
• DSP161	16	Extraction of PCDD/PCDF from Water for Methods 1613, 8290, and 551
• DSP260[1]	5	SLICC III - Acid Aluminum
• DSP270[1]	2	Preparing, Cleaning, and Activating Column Packings for Cleanup
• SMC114	2	Creating the TLI Chain of Custody
• SMC120	1	Sample Storage and Handling
Microseeps, Inc. SOPs:		
• AM18.01[1]		Analysis of C_1 - C_4 Hydrocarbons in Water
• Addendum[1]		Addendum to Microseeps Method AM18.01
B&R Environmental SOPs:		
• CT-05	0	Database Records and Quality Assurance

1 This SOP is considered to be confidential and proprietary. It will be submitted to U.S. EPA Region 5 for review under separate cover.

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1001

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

Revision history:

Number	Date
4	5/3/99
3	7/22/94
2	9/16/87
1	7/31/86

Written by: Harry Romberg
Harry Romberg, QA Officer
Date: 5-3-99

Approved by: Kathy Kreps
Kathy Kreps, Laboratory Director
Date: 5-6-99

Controlled Document
No. 20 Assigned to: Tetra

Table of Contents

1. INTRODUCTION AND SCOPE 3

1.1 PURPOSE 3
1.2 SCOPE 3

2. PROCEDURES 3

2.1 WORD PROCESSING FORMAT 3
2.2 INITIATING AN SOP OR METHOD 3
2.3 REVISING AN SOP 5

3. ELEMENTS OF AN SOP/METHOD 5

3.1 ELEMENTS 5
3.2 SAVING THE DOCUMENT 8
3.3 AFTER COMPLETION OF THE DRAFT SOP 8

1. Introduction and Scope

1.1 Purpose

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

1.2 Scope

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

1.2.2 Methods are considered to be technical analytical procedures and are generally derived from EPA or other analysis methods. 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.

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

2. Procedures

2.1 Word Processing Format

2.1.1 All new SOPs are written in WORD 6 format (or higher when accepted by QA). Older versions of SOPs may be written in WORD 2 or XyWrite, however, these formats should be updated to meet the current format as they are revised.

2.1.2 Flow charts may either be photocopied from methods, scanned and inserted into the document electronically or prepared from programs such as Flow 4 (Patton & Patton Software) and inserted into the document electronically. Any of these electronic files should be kept together with the electronic version of the document to facilitate its modification and inclusion into future updates.

2.2 Initiating an SOP or Method

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

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. For tracking and control purposes, all will be assigned a number by the QA Officer or designee. This number will begin with the letters LTL- (such as this

SOP, LTL-1001). A number should be obtained from QA before the author begins writing the SOP but if this is not possible, a number must be obtained before the SOP can be turned in for review.

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

2.2.4 The author may then use the appropriate Word template (SOPhead, Iorgtemp or Orgtemp) from the [File][New] menu presented in Word. Hardcopies of these formats are not included in this SOP but may be accessed by the reader using the above means. These formats may change somewhat without updating of this SOP but if the author accesses the template from the laboratory network in this manner, the latest version will be automatically used.

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

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

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

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

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

2.2.7 Draft versions of SOPs should be worked on in the "projects" drive (p: on most computers). They should then be located in the p:\sop directory under the subdirectory most relevant to the areas addressed by the SOP. For instance, in writing this SOP, it was

stored in p:\sop\qa_sops. Metals SOPs would be worked on in the p:\sop\metals subdirectory. QA will transfer the final document to the appropriate location for permanent storage and archival in order to maintain copies of all of the appropriate revisions.

2.3 Revising an SOP

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

3. Elements Of An SOP/Method

3.1 Elements

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

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

3.1.3 Methods contain the appropriate cover (title and revision number as on the cover of this SOP), header information, table of contents, introduction and scope, equipment, reagents, specific operating procedures, calibration and quality control (including corrective actions), and any appropriate appendices. They should also include data package assembly information and run sequences. Appendices should include preparation of standard solutions, a Method QC Table and a procedural flow chart.

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

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

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

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

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

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

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

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

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

3.1.11 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.

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

3.1.13 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.

3.1.14 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.

3.1.15 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.

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

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

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

3.3 *After Completion of the Draft SOP*

3.3.1 After the document has been written to the satisfaction of the author, it should be first passed to the department/division manager (unless that's who wrote it) for technical review. From there, the QA Officer and Lab Director will review and approve the document. QA will distribute all approved and signed documents. An unapproved (fully signed) SOP or Method document is not considered official. Other details of the document tracking process are discussed in that SOP (LTL-1002).

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
4	8/4/99

Revised by: Harry Romberg Date: 8-4-99
Harry Romberg, Quality Assurance Officer

Approved by: Kathy Kreps Date: 8-4-99
Kathy Kreps, Laboratory Director

Controlled Document

No. 20 Assigned to: Tetra

Table of Contents

1. INTRODUCTION AND SCOPE 3

1.1 DESCRIPTION 3
1.2 SCOPE 3

2. DEFINITIONS 3

3. RESPONSIBILITIES 4

3.1 ANALYST 4
3.2 SUPERVISOR 4
3.3 QA 5
3.4 TRAINER 5

4. OPERATION PROCEDURES 5

4.1 RECOGNITION OF EXPERIENCE AND TRAINING 5
4.2 DEMONSTRATION OF CAPABILITY TO PERFORM ANALYSIS 7
4.3 ONGOING DEMONSTRATION OF PERFORMANCE 9

5. REFERENCES 9

APPENDIX A 10

RECOGNITION OF EXPERIENCE AND TRAINING FORM 10

APPENDIX B 12

DEMONSTRATION OF CAPABILITY TO PERFORM ANALYSIS FORM 12

APPENDIX C 14

EXAMPLE TRAINING CHECKLISTS 14

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. *This practice also usually includes the completion of training checklists for the task for which the analyst is being trained.*

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.

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

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 have 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 Competency may also be demonstrated by successful analyses of any combination of at least 3 each of at least two types of spiked QC samples (blank spikes, matrix spikes or lab control samples). These may be documented on the forms in Appendix B and the training forms in Appendix C but a summary must accompany the certifying form which either includes or provides reference to the data.

4.1.6 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.

4.1.7 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, 4.1.4 *or 4.1.5.*

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 Alternatively, training checklists for many tasks have been developed. In the case of analytical duties, these may include reference to the completion of P&A or PE studies or individual QC analyses as previously discussed. Completion of one of these training checklists (including appropriate signatures) is considered demonstration of training. However, PE or P&A results or other appropriate documentation should also accompany the training checklist as demonstration of competency.

4.2.11 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.

4.2.12 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

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

compdemo.doc/rev.2 12/13/95

Appendix C

Example Training Checklists

Laucks Testing Labs

Pesticide/Herbicide GC Semivolatile Analyst Training Verification Checklist

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

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

_____

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

Laucks Testing Labs
GC Volatile Analyst Training Verification Checklist

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					
1	Able to use Standards Log				
2	Able to use Instrument Run Logs				
3	Able to use Instrument Maintenance Logs				
Methods					
4	Has read and understands SOPs for all applicable methods				
	List Method(s): _____				
5	Has read and understands EPA & State Methods				
	List Method(s): _____				
6	Has read and understands appropriate sections of GC Training Manual				
Instrument Operation/Maintenance					
7	Knows location and use of Instrument Manuals				
8	Knows basic GC theory				
9	Able to use GC Control Pad to set temperature program				
10	Able to use Autosampler Control Pad to set injection program				
11	Able to change syringe, septa & injection port liner				
12	Able to trim/change columns & perform leak check				
13	Able to measure and set carrier and makeup gas flows				
14	Able to bake column/injectors/detectors				
15	**NON-ROUTINE:** *Able to change detectors*				
16	**NON-ROUTINE:** *Able to perform total system cleaning*				
Analytical Performance					
17	Able to prepare standards & pass standard QC acceptance criteria				
18	Able to analyze RTM standard and set up elution range				
19	Able to analyze and generate acceptable calibration curve				
20	Able to analyze CCVs and apply QC acceptance criteria				
21	Applies acceptance criteria for surrogates and spikes				
22	Able to set up analytical runs & acquire data				
23	Able to get information on samples/analyses (test codes, MDLs, etc.)				
24	Able to quantitate an analytical batch (standards, CCVs, QC & samples)				
25	Knows reanalysis and reextraction criteria				
26	Able to perform sample dilutions (obtaining linear results)				
27	Knows correct reporting limits for method(s)				
28	Knows corrective action & documentation for out of control QC events				
29	Able to produce a data package (In-house and SW-846)				
Method Validation *(complete one or more of the following)*					
30	Has successfully analyzed four P&A samples				
31	Has successfully analyzed two PE samples				
32	Has successfully analyzed three each of two types of QC samples				

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

_____

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

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
GC/MS 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 (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/MS theory				
9	Able to use GC Control Pad to set temperature program				
10	Able to use Autosampler Control Pad to set analysis program				
11	Able to change syringe, septa & injection port liner				
12	Able to trim/change columns & perform leak checks				
13	Able to measure and set carrier and makeup gas flows				
14	Able to bake column/injectors/detectors				
15	**NON-ROUTINE:** *Able to clean total system including P&T and autosampler*				
16	**NON-ROUTINE:** *Able to clean source*				
Analytical Performance					
17	Able to prepare daily standards				
18	Able to inject BFB				
19	Able to tune instrument to meet method specifications				
20	Able to analyze and generate acceptable calibration curve				
21	Able to analyze CCVs and apply QC acceptance criteria				
22	Applies acceptance criteria for surrogates and spikes				
23	Able to set up analytical runs (CLP & non-CLP) & acquire data				
24	Able to get information on samples/analyses (test codes, MDLs, etc.)				
25	Able to quantitate an analytical batch(standards, CCVs, QC & samples)				
26	Knows how to confirm detection of analytes (rt, spectra confirmation)				
27	Knows reanalysis criteria				
28	Able to perform sample dilutions (obtaining linear results)				
29	Knows correct reporting limits for method(s)				
30	Knows corrective action & documentation for out of control QC events				
31	Able to produce a data package (In-house, CLP and SW-846)				
Method Validation *(complete one or more of the following)*					
32	Has successfully analyzed four P&A samples				
33	Has successfully analyzed two PE samples				
34	Has successfully analyzed three each of two types of QC samples				

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

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

Laucks Testing Labs
Metals ICP/MS Analyst Training Verification Checklist

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

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

_____

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

Laucks Testing Labs
Mercury Analyst Training Verification Checklist

Analyst Name:		Date:	Trainer:	Supervisor:	Analyst:
Documentation					
1	Able to enter standards in the logbook				
2	Able to generate an instrument run log				
3	Able to make instrument maintenance logbook entries				
Methods					
4	Has read the SOPs for all applicable methods				
	List SOP(s): LTL-7501				
5	Has read and understands EPA & State Methods				
	List Method(s): 254.2, 7470,7471				
6	Has read the appropriate sections of the FIMS 400 Instruction Manual				
Instrument Operation/Maintenance					
7	Knows location and use of Instrument Manuals				
8	Knows basic theory				
9	Able to use the software to create a method				
10	Able to use the software to set up the autosampler table				
11	Able to change the peristaltic pump tubing				
12	Able to clean the spectrophotometer windows				
Analytical Performance					
18	Able to prepare standards & pass standard QC acceptance criteria				
19	Able to analyze and generate an acceptable calibration curve				
20	Able to analyze CCVs and apply QC acceptance criteria				
22	Able to set up an analytical run & acquire data				
3	Able to copy the analytical run file from the hard drive to the network				
24	Able to quantitate an analytical batch (standards, CCVs, QC & samples)				
25	Knows reanalysis and redigestion criteria				
26	Able to perform sample dilutions				
27	Knows correct reporting limits for method(s)				
28	Knows corrective action & documentation for out of control QC events				
29	Able to correct the data file in preparation for producing a data package				
Method Validation *(complete one or more of the following)*					
30	Has successfully analyzed four P&A samples				
31	Has successfully analyzed two PE samples				
32	Has successfully analyzed three each of two types of QC samples				

This is to certify that _____ has been analyzing samples by the methods stated above 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
Sample Custodian Training Verification Checklist

Employee Name:		Date:	Trainer:	Supervisor:	Trainee:
Documentation					
1	Able to use Chain of Custodies for received samples				
2	Able to use Sample Receipt, Discrepancy and Preservation Forms				
3	Able to use Internal Chain of Custody Logs				
4	Able to use Chain of Custodies for subcontracted sample analysis				
Methods					
5	Has read and understands SOPs for all applicable job responsibilities				
	List SOP(s): _____				
Sample Receipt Process					
6	Knows how to receive samples				
7	Knows SAM basics, including the use of function keys				
8	Able to create client information				
9	Able to create workorders				
10	Able to enter samples in fraction screen				
11	Able to use global jobs and client jobs				
12	Able to create and use SDGs				
13	Able to complete bottle log in SAM				
14	Knows how and where to store samples once received				
15	**NON-ROUTINE:** *Able to create global jobs and client jobs*				
Additional Responsibilities					
16	Able to prepare, package and ship samples to subcontract lab				
17	Able to preserve and stock bottles				
18	Able to order bottles				
19	Able to complete bottle orders				
20	Able to ship bottle orders by UPS				
21	Able to ship bottle orders by US Mail				
22	Able to ship bottle orders by Fed Ex				
23	Able to ship bottle orders by local courier				
24	Knows how to dispose of soils and document disposal				
25	Knows how to neutralize and dispose of waters and document disposal				
26	Knows and uses appropriate PPE for handling samples				
27	Able to perform sample splitting				
28	Able to perform sample preservation for volatiles methods				
Validation *(complete all of the following)*					
29	Has successfully logged in samples for one month				
30	Has successfully prepared and shipped bottles by all carriers				
31	Has successfully created client and/or global jobs				
32	Has successfully disposed of samples and documented disposal				

This is to certify that _____ has been a sample custodian in the sample receiving department and has demonstrated competency at the preceeding tasks.

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

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1012

Title: Solvent QC Monitoring for Trace Residue Analysis

Revision history:

Number	Date
2	4/27/99
1	4/10/91

Written by: Harry Romberg, Quality Assurance Officer Date: 4-29-99

Reviewed by: Monica Carr, Organics Division Manager Date: 4/29/99

Approved by: Kathy Kreps, Laboratory Director Date: 4/29/99

Controlled Document

No. 20 Assigned to: Tetra

Table of Contents

1. INTRODUCTION AND SCOPE 3

1.1 DESCRIPTION 3

2. EQUIPMENT LIST AND REAGENTS 3

2.1 EQUIPMENT AND REAGENTS 3

3. SAFETY PRECAUTIONS AND WASTE DISPOSAL 3

3.1 SAFETY PRECAUTIONS 3
3.2 WASTE DISPOSAL 3

4. OPERATION PROCEDURES 3

4.1 SEQUESTERING OF SOLVENT 3
4.2 INITIATION, DATA HANDLING AND RECORD MAINTENANCE 4
4.3 SOLVENT ANALYSIS 5
4.4 DATA PACKAGE ORGANIZATION 7

APPENDIX I 8

SOLVENT ACCEPTANCE CRITERIA 8

1. Introduction and Scope

1.1 Description

1.1.1 The purpose of this SOP is to define the method(s) used to check and document the purity of the major solvents used for trace residue analysis at Laucks. The solvents being tested are methylene chloride, acetone, and hexane. Specific techniques and equipment used for operations such as concentration and solvent exchange are not addressed in this document.

2. Equipment List and Reagents

2.1 Equipment and Reagents

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

3. Safety precautions and Waste Disposal

3.1 Safety Precautions

3.1.1 Typical precautions should be taken when handling any solvent. Some, such as methylene chloride or freon are not flammable, but others, such as acetone and hexane are and should be treated with extreme caution. Long term health effects of solvent contact are often unknown but generally unfavorable. Breathing of ANY solvent vapor should be minimized, as should any direct skin contact, by working in a well ventilated area (in or near a hood if necessary) and by using the provided gloves and, if necessary or desired, respirator masks.

3.2 Waste Disposal

3.2.1 All waste solvents should be disposed in the appropriate waste solvent container, never in the sink or in combination with aqueous liquids.

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

4. Operation procedures

4.1 Sequestering of Solvent

4.1.1 A new lot of acetone or methylene chloride must be sequestered by the supplier and the checking process initiated at least four weeks prior to using up the last of the previous lot

of that solvent. A lot is defined as a batch of solvent with the same manufacturers lot number. This must be done in order to ensure that the lot has been released for analytical use BEFORE the remainder of an acceptable lot has been used up. If any solvent has failed, a second bottle may be tested for the failed parameter(s) in order to ensure the failure was not due to laboratory contamination. Failure of the second test is grounds to reject that lot for use in the laboratory.

4.1.2 When a lot has been formally designated as acceptable, enough should be ordered to last approximately 2 months in order to minimize the frequency of testing necessary. Typically, the supplier will ship 10 cases of a sequestered lot of methylene chloride each week and acetone as requested. No more than 4 months supply of methylene chloride will ever be ordered, as typical methylene chloride recommended shelf-life is 6 months.

4.1.3 Alternatively, since it is unlikely that any lot will fail and to eliminate the time between acceptance and delivery, an appropriate supply (as defined above) may be ordered and sequestered at the laboratory for analysis. This is commonly done for hexane since we do not consume large quantities of this solvent. If said lot fails, however, the lot must be returned to the supplier and a new lot tested immediately. This lot MUST be kept separate from the current stock and very clearly marked so that it is not inadvertently used prior to acceptance. This distinction is the responsibility of the Extractions Supervisor. All solvent deliveries must be immediately reported to the Extractions Supervisor or designated alternate in order that this distinction be made.

4.2 Initiation, Data Handling and Record Maintenance

4.2.1 The Extractions supervisor or designated representative initiates the checking process. For any month in which any extractions solvent or other reagent QC is performed, a LIMS workorder is created. When a bottle from a new, previously untested lot of solvent is received, a sample ID number is assigned from the next available ID in the workorder. This "fraction" ID must contain the manufacturer, lot number, solvent, and tests (test codes) to be performed.

4.2.2 The three solvents which we specifically check in the extractions lab are methylene chloride, acetone, and hexane. The test codes used for these purposes are MECLAC, MSQCCK, and PXQCCK. These codes are used **only** for testing solvents and for nothing else. Other checks, such as sodium sulfate or Florisil should use the test code appropriate for the analysis for which the material will be used (8081, 8270, etc.)

4.2.2.1 Methylene chloride is checked for acidity, semivolatiles and pesticides/PCBs. The respective test codes assigned to these analyses are MECLAC, MSQCCK, and PXQCCK.

4.2.2.2 Acetone is checked for semivolatiles and pesticides/PCBs. The respective test codes assigned to these analyses are MSQCCK, and PXQCCK.

4.2.2.3 Hexane is checked only for pesticides. The respective test code assigned to this analysis is PXQCCK.

4.2.3 When testing has been completed, the lot will be officially designated as acceptable or failed by the QA Officer or Technical Director. This will be done by initialing the final report and sending a copy to the Extractions supervisor. Any lot will be considered acceptable which meets the criteria specified in Appendix I. The Extractions supervisor should be certain that a lot has been designated as acceptable prior to using it and should take whatever actions are necessary to ensure prompt analysis and acceptance before the last of the acceptable solvent has been used.

4.2.4 The data and report files will be maintained by the QA Officer. After all of the QC in the month has been closed and signed off, the data file and acceptance sheets will be filed with the regular workorder files.

4.3 Solvent Analysis

4.3.1 Methylene Chloride Acidity

4.3.1.1 0.01 N NaOH - To a 100 mL. volumetric flask, add 10 mls. of .1000 N sodium hydroxide from the buret of standardized NaOH in the Inorganics lab. Fill to the volumetric mark with deionized water, stopper, and mix very well. It takes several inversions of the flask to properly mix the solution (at least 10). This solution should be prepared immediately prior to analysis.

4.3.1.2 Neutral ethanol - Add 25 mls. of denatured ethanol to an Erlenmeyer flask. Add 2 or more drops of phenolphthalein indicator solution (1 gm. phenolphthalein/100 mls. ethanol). With a Pasteur pipet, add the .01 N NaOH solution dropwise until the ethanol turns slightly pink. Hold the flask against a white background to enhance the color. This solution should be prepared immediately prior to analysis

4.3.1.3 Add 25 mls. of the methylene chloride to be checked to the flask containing neutralized ethanol. Swirl. Do not shake too vigorously so that CO_2 from the air will not acidify the ethanol and cause a fading endpoint.

4.3.1.4 Add 900 uL of the 0.01 N NaOH. Swirl to mix well.

4.3.1.5 If the resulting color is pink, the methylene chloride passes (is not acidic). If it does not turn pink, it should be retested, preferably from a second bottle. If it fails

a second time, it should be rejected or used only for cleaning. Failing solvent should NEVER be used for extraction purposes.

4.3.1.6 A "PASS" or "FAIL" is entered into the SAM report under the associated regular SAM test code, MECLAC. If the solvent fails, residue analysis SHOULD NOT be performed until a suitable acceptable lot is determined. The Extractions supervisor should see that any such failing lot has been terminated in SAM. Data and the report, however, should still be submitted to the QA Officer.

4.3.2 Residue Checks

4.3.2.1 The residue checks are performed for EPA CLP Target Compound List (TCL) components for both pesticides/PCBs and semivolatiles (ABNs) as is appropriate for the solvent being checked.

4.3.2.2 In all cases, varying amounts of the appropriate solvent is concentrated to 1 mL. in a Kuderna-Danish concentrator. No splitting of the concentrate occurs. Surrogates are not added.

4.3.3 Methylene Chloride - $MeCl_2$ is used for both ABN and pesticide/PCB analyses. A separate 400 mL. concentration is done for each analysis.

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

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

4.3.4 Acetone - Acetone is used for both ABN and pesticide/PCB analyses. A separate 150 mL. concentration is done for each analysis.

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

4.3.4.2 For the ABN analysis, the acetone is blown down to near dryness with nitrogen and brought up to 1 mL. with $MeCl_2$ and submitted for analysis.

4.3.5 Hexane - Hexane is used only in pesticide analysis. It will be concentrated 25 mls. to 1 mL. as stated and submitted for TCL pesticide/PCB analysis.

4.3.6 Acceptance criteria are compiled in Appendix I and are based on the appropriate amounts of solvent concentrated to 1 mL. final volume. They are derived from Laucks reporting

limit criteria for acceptable blanks. The LIMS report indicates the acceptance level, the level found and signifies whether the detected level (if any) passes (OK) or fails (FAIL).

4.4 Data Package Organization

4.4.1 A copy of the signed acceptance form along with the raw data is retained by QA under the assigned laboratory number. The Extractions supervisor keeps a photocopy of the signed approval sheet in a file in the extractions lab.

Appendix I

Solvent Acceptance Criteria

Solvent Acceptance Criteria

Semivolatile Compounds	Total ng in 1 mL.
Phenol	5000
bis(2-Chloroethyl) ether	5000
2-Chlorophenol	10000
1,3-Dichlorobenzene	5000
1,4-Dichlorobenzene	5000
1,2-Dichlorobenzene	5000
2-Methylphenol	5000
2,2'oxybis(1-Chloropropane)	5000
4-Methylphenol	5000
N-Nitroso-di-n-propylamine	5000
Hexachloroethane	5000
Nitrobenzene	5000
Isophorone	5000
2-Nitrophenol	10000
2,4-Dimethylphenol	5000
bis(2-Chloroethoxy)methane	5000
2,4-Dichlorophenol	10000
1,2,4-Trichlorobenzene	5000
Naphthalene	5000
4-Chloroaniline	5000
Hexachlorobutadiene	5000
4-Chloro-3-methylphenol	5000
2-Methylnaphthalene	5000
Hexachlorocyclopentadiene	5000
2,4,6-Trichlorophenol	10000
2,4,5-Trichlorophenol	10000
2-Chloronaphthalene	5000
2-Nitroaniline	5000
Dimethylphthalate	25000
Acenaphthylene	5000
2,6-Dinitrotoluene	5000
3-Nitroaniline	5000
Acenaphthene	5000
2,4-Dinitrophenol	10000
4-Nitrophenol	10000
Dibenzofuran	5000
2,4-Dinitrotoluene	5000
Diethylphthalate	25000

Semivolatile Compounds	Total ng in 1 mL.
4-Chlorophenyl-phenyl ether	5000
Fluorene	5000
4-Nitroaniline	10000
4,6-Dinitro-2-methylphenol	10000
N-nitrosodiphenylamine	5000
4-Bromophenyl-phenylether	5000
Hexachlorobenzene	5000
Pentachlorophenol	10000
Phenanthrene	5000
Anthracene	5000
Carbazole	5000
Di-n-butylphthalate	25000
Fluoranthene	5000
Pyrene	5000
Butylbenzylphthalate	25000
3,3'-Dichlorobenzidine	10000
Benzo(a)anthracene	5000
Chrysene	5000
bis(2-Ethylhexyl)phthalate	25000
Di-n-Octylphthalate	25000
Benzo(b)fluoranthene	5000
Benzo(k)fluoranthene	5000
Benzo(a)pyrene	5000
Indendo(1,2,3-cd)pyrene	5000
Dibenz(a,h)anthracene	5000
Benzo(g,h,i)perylene	5000

Solvent Acceptance Criteria

Pesticide/PCB Compounds	Total ng in 1 mL.
alpha-BHC	5
beta-BHC	5
delta-BHC	5
gamma-BHC	5
Heptachlor	5
Aldrin	5
Heptachlor epoxide	5
Endosulfan I	5
Dieldrin	10
4,4'-DDE	10
Endrin	10
Endosulfan II	10
4,4'-DDD	10
Endosulfan sulfate	10
4,4'-DDT	10
Methoxychlor	50
Endrin ketone	10
Endrin aldehyde	10
alpha-Chlordane	5
gamma-Chlordane	5
Toxaphene	500
Aroclor-1016	100
Aroclor-1221	200
Aroclor-1232	100
Aroclor-1242	100
Aroclor-1248	100
Aroclor-1254	100
Aroclor-1260	100

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-4002

Title: **Electronic Sample Entry and Log-In**

Revision history:

Number	Date
6	3/18/99
5	6/19/96
4	12/28/95
3	4/05/95
2	6/05/90
1	4/24/90

Revised by: Pam Johnson — Date: 3/25/99
Pam Johnson, Sample Control

Approved by: Kathy Kreps — Date: 3-25-99
Kathy Kreps, Laboratory Director

Approved by: Harry Romberg — Date: 3-25-99
Harry Romberg, QA Officer

Controlled Document

No. 20 **Assigned to:** TETRA

TABLE OF CONTENTS

1. INTRODUCTION AND SCOPE 4

1.1 INTRODUCTION 4
1.2 SCOPE 4

2. EQUIPMENT LIST 4

3. SAFETY PRECAUTIONS 4

3.1 SAMPLE HANDLING 4

4. OPERATION PROCEDURES 5

4.1 SAMPLE RECEIPT 5
4.2 SAMPLE LOG-IN 7
4.3 SPECIAL DOCUMENTATION PROCEDURES FOR CLP SAMPLES 11
4.4 ASSIGNMENT OF SDG NUMBERS 12
4.5 ASSIGNMENT OF LAB QUALITY CONTROL SAMPLES 12
4.6 COMPLETION OF SDG RECORDS 13
4.7 SAMPLE STORAGE 16
4.8 STORAGE LOCATIONS 17
4.9 DETERMINATION OF TESTS 18
4.10 ELECTRONIC TRANSMITTAL OF SAMPLE AND TEST REQUEST RECORDS 18
4.11 GENERATION OF INTERNAL CHAIN-OF-CUSTODY (COC) 19
4.12 SAMPLE BREAKAGE 20
4.13 SPECIAL CIRCUMSTANCES 20

APPENDIX 1 21

SAMPLE RECEIPT VERIFICATION STAMP 21

APPENDIX 2 22

NON-CLP SAMPLE RECEIPT LOG 22

APPENDIX 3 24

CLP SAMPLE RECEIPT LOG 24
SUPPLEMENTAL SAMPLE RECEIPT LOG 24

PAGE 3 OF 3 27

APPENDIX 4 28

SECURE STORAGE CUSTODY LOG 28
ORGANIC EXTRACTIONS CUSTODY LOG 28

APPENDIX 5 31

LAUCKS TESTING LAB pH LOG FORM 31

APPENDIX 6 33

LAUCKS TESTING LAB SAMPLE SPLIT SHEET 33
APPENDIX 7 ..... **35**
REQUIRED CONTAINERS/VOLUMES/PRESERVATION/HOLDING TIMES 35
APPENDIX 8 ..... **51**
CHECKLIST FOR ANALYSES WITH SHORT HOLDING TIMES 51

1. INTRODUCTION AND SCOPE

1.1 Introduction

1.1.1 This procedure is a description of sample receipt, sample log-in, and sample tracking when samples are logged into the laboratory's Laboratory Information Management System (LIMS). The collection of programs and procedures which comprise the LIMS is called "SAM." References made to SAM in this SOP are references to this collection of programs and procedures.

1.1.2 Sample entry must be performed in a timely fashion to allow tests with short holding times to be started immediately. Accuracy in the recording of sample IDs, in marking samples with lab numbers, and in checking for consistency of all records is of utmost importance.

1.2 Scope

1.2.1 All samples received by the laboratory are logged using the following procedures.

2. EQUIPMENT LIST

Lab coat
Disposable gloves
Respirator, dust mask
3M desk cleaner, broom, dustpan, mop
Spatula
Waterproof labeling gun
PC work station linked to SAM

3. SAFETY PRECAUTIONS

3.1 Sample Handling

3.1.1 Samples received at the laboratory can potentially be contaminated with toxic materials. Reasonable caution must be exercised at all times when handling these samples. Such precautions include wearing a lab coat at all times, using gloves, using a hood (located in Inorganics) to perform operations when necessary (strong odors present, etc.), and wearing a respirator or dust mask if fumes or dust are generated.

3.1.2 Cleanliness and neatness are of utmost importance. All spills and condensation from wet sample containers must be cleaned up immediately. This will help to alleviate accidental sample breakage and protect others from possible contact with contaminated work areas.

3.1.3 When wearing gloves, be certain to remove them when opening the cooler or lab doors and when answering the phone. The gloves which protect the sample 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.

3.1.4 A dust mask is worn when pouring dry packing material such as vermiculite into the garbage.

4. OPERATION PROCEDURES

4.1 Sample Receipt

4.1.1 Samples may be received by client delivery, over the front counter, via UPS, courier services, by various 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. **NON-CLP** Sample Receipt Log (Appendix 2) is for Laucks **NON-CLP** log-in procedure. If requested a client provided receipt form may be substituted for the Laucks sample receipt log.

4.1.5 Put on gloves, open the coolers (in the hood if necessary), and note whether custody seals are present and, if so, intact. **Affix one of the intact custody seals on the sample receipt log.** 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 either the enclosed temperature blank or at least 3 separate containers taken randomly from different locations in EACH cooler must be checked for temperature with the infrared thermometer. The temperatures are recorded on the Supplemental Sample Receipt Log (Appendix 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.

4.1.11 After all samples are arranged then check consistency between the COC and the sample labels for Sample IDs, dates and times on each sample container.

4.1.12 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).

4.1.13 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.

4.1.14 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.

4.1.15 All sample container marks (including ID's, dates and times) 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 Non-CLP Sample Receipt Log (Appendix 2) or the CLP Sample Receipt Log (Appendix 3) and reported to the client.

4.1.16 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
940149                                                                  SAMPS
 KEYS    ST  ORDER # 94-01-496                  RECEIVED 01/12/94        2
9401485  WR                                     DATE DUE 01/26/94
9401486  TR   CLIENT 1_LH_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 L.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 NL 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.

4.2.4 All required data entry fields are in inverse video (highlighted) on the screen, but fields for purchase order numbers, project name or number, and point of contact should also be completed, if the information is known.

4.2.5 All sample IDs, dates of collection, and dates of receipt are recorded in the FRAC program for the 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.

```
94041                Laucks Testing  FRACTION DESCRIPTION 04/05/94 13:36:36
   KEYS                 ORDER# 94-04-191/WR CON MARKB          DEPTS/TESTS
9404176-12A             CLIENT PETER_PAN    CAT                              HIST1
9404176-13A           WRITTEN 04/05/94       BY HOLLYW                      HIST2
9404176-13B
9404176-13C                DASH # 02 A CATEGORY
9404176-13D           SAMPLE ID Mahi Mahi Lot #X3159 (#2)
9404176-14A           COLLECTED 04/05/94            QA/QC    MAT F
9404176-15A              STORED Analyst/Freezer    RECVD 04/05/94 SC    2
9404176-15B                                          DUE 04/15/94
9404176-15C           JOB HIST2  FL    QC?          QUOTE     75.00
9404176-15D          NAME Histamine                   SDG#          StID#
9404176-16A          TESTS:DEP/CAT/NAME/D/R
9404176-17A             HISTAM
9404176-17B
9404176-17C
9404176-17D
9404176-18A          -SAMPLE AND WORK DESCRIPTION-
9404191-01A           This job code is for any additional samples.
9404191-02A
                                                                      CL NL SL J1
 F5=Result,Price  F6=Lookup,NextJob,StateID   F10=More   Shft-Fn=Fn Help
```

FRAC Screen

4.2.6 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.

4.2.7 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

4.2.8 Each sample or set of samples is assigned a unique identifying work order number, generated by the Laboratory Information Management System (LIMS), on receipt. This unique number consists of 7 numerical characters, such as 9007215. In this example, the work order was initiated in 1990 (90), in the month of July (07) and was the 215th such work order that month (215). If more than 999 workorders are generated in any month, the 5th digit is replaced by successive letters of the alphabet (A-Z).

4.2.9 If necessary, more than one fraction may be created for a sample (generally, this is related to billing issues - when one analysis is discounted in price and another is not, for instance), but they will all bear the same fraction number and be differentiated by an automatically-assigned letter suffix. For instance, if sample 9004001-01 had 3 fractions, purely for internal accounting reasons, the three fractions would be identified as:

9004001-01A
9004001-01B
9004001-01C

4.2.10 The person performing log-in needs to be aware of this effect, but it has no impact on sample identification within the lab, on sample tracking, or on the sample number placed on the bottles/jars. In the above example, all bottles submitted for this sample would be marked 9004001-01.

4.2.11 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.

4.2.12 This information must be recorded in the "Bottles" computer tracking program under each workorder and the workorder-unique bottle identifier will be printed in the bottom left corner of each bottle label (which also contains the workorder number and the sample number) before the bottle label is affixed to each individual bottle.

4.2.13 **ALERT:** Each SAM work order can accommodate up to 57 fractions and no more. Each work order must allow sufficient fraction space for later changes or additions. Therefore, no more than 50 samples should be logged into any single work order. If, for administrative reasons, some or all of the samples consist of more than one fraction, then no more than 50 <u>fractions</u> can be logged. Should the submittal consist of more than 50 samples, or more than 50 fractions, initiate additional work orders as required. Cross-reference the work order numbers, so that all samples submitted together can be reported

together to the client. You can perform this cross-reference manually (by noting on accompanying documents "See Also [Work Order Number]) or you can make appropriate comments in the Work Order Comment field (F2). To the degree possible, make sure that multiple work orders which represent one complete project in the client's mind are created sequentially, with no other unrelated work order numbers intervening.

4.2.14 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.

4.2.15 **ALERT:** Each fraction will accommodate only 27 tests. If more than 27 analyses are required on any sample, additional fractions should be made (i.e. -1A, -1B, -1C, etc.).

4.2.16 **ALERT:** The 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

4.3.2 CLP Sample Receipt Log and the Supplemental Sample Receipt Log are CLP-specific sample login sheets. For each cooler received a CLP Sample Receipt Log Form and a Supplemental Sample Receipt Log must be completed. This form takes the place of the NON-CLP Sample Receipt Log (Appendix 2). Copies of these forms may be found in Appendix 3.

4.3.3 Complete the header information requested at the top of the forms. Use multiple pages if necessary.

- date received
- time received
- client name
- SDG #
- COC # (if available)
- sample log-in date
- work order #
- client project
- airbill number (if available)
- and initials of the person logging in the samples.

4.3.4 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.

4.3.5 Since the extractable fractions will be transferred to the extractions lab, a Secure Storage Custody Log must be completed, and the samples are held on 8C in the WO1 walk-in cooler (extractions hold shelf) pending pick-up by extractions personnel. Specifics of the Storage Custody Log is outlined in the Chain-Of-Custody SOP located in the SOP manual.

4.4 Assignment of SDG numbers

4.4.1 The SDG name is assigned by sample control and is usually based on client name or project name followed by sequential numbering.

4.5 Assignment of lab quality control samples

4.5.1 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.

4.5.2 Note in the **SAM SDG** records which sample is QC-assigned.

4.5.2.1 This information is pulled into SDG from the FRAC program when the SDG is created.

4.5.2.2 On the FRACTION screen, an X is placed in the QC field. This will print out next to the sample and indicate that it is an assigned QC sample.

4.5.2.3 Write a department comments message for whichever department (EX, MS, and/or GC) should be notified.

4.6 Completion of SDG records.

4.6.1 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.

4.6.2 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
COM      SDG Group : CDM10        Date Due: 03/13/93        Created:  02/19/93
  KEYS   Fraction  : E SPVMITO                              Updated:  02/19/93
BOGD5G   Project   : CDM (EDB)              Client:
BOGD5I   SAS Number:                Case Number:            Max. Samps:
BOGD6G
BOGD6I    Work   Samp                                                Associated
BOGD7G    Ord    Num  QC            Client ID          Mat TS pH      Blank
BOGD7I
BRPTSS
CANONS   9302598 17     07-MW-005-B-000-F                W
CB2SFV   9302598 20     07-MW-007-B-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 : CDM10        Date Due: 03/13/93        Created:  02/19/93
  KEYS     Fraction  : E SPVMITO                              Updated:  02/19/93
BOG05G     Project   : CDM (EDB)              Client:
BOG05I     SAS Number:              Case Number:            Max. Samps:
BOG06G
BOG06I      Work  Samp            Date
BOG07G      Ord   Num   VTSR    Collected
BOG07I
BRPTSS
CANONS     9302598 17 02/16/93 02/13/93
CB2SFV     9302598 20 02/16/93 02/13/93
CB3-3V
CBSFXS
CDM10E
CDM10G
CDM10I
CDM10P
CDM10S
CDM10V
CDM11E
CDM11G
                                                                   CL NL SL J1
5=Lookup,Copy,Print  6=Toggle      F10=More    Shft-Fn=Fn Help
```

SDG, Screen 2

```
                                   SDG Database
COM        SDG Group : CDM10        Date Due: 03/13/93        Created:  02/19/93
  KEYS     Fraction  : E SPVMITO                              Updated:  02/19/93
BOG05G     Project   : CDM (EDB)              Client:
BOG05I     SAS Number:              Case Number:            Max. Samps:
BOG06G
BOG06I      Work  Samp                        Fractions
BOG07G      Ord   Num   EDB
BOG07I
BRPTSS
CANONS     9302598 17    X
CB2SFV     9302598 20    X
CB3-3V
CBSFXS
CDM10E
CDM10G
CDM10I
CDM10P
CDM10S
CDM10V
CDM11E
CDM11G
                                                                   CL NL SL J1
5=Lookup,Copy,Print  6=Toggle      F10=More    Shft-Fn=Fn Help
```

SDG, Screen 3

```
                                   SDG Database
CDM       SDG Group : CDM10        Date Due: 03/13/93        Created:  02/19/93
  KEYS    Fraction  : E SPVMITO                             Updated:  02/19/93
BOGD5G    Project   : CDM (EDB)            Client:
BOGD5I    SAS Number:              Case Number:            Max. Samps:
BOGD6G
BOGD6I
BOGD7G
BOGD7I
BRPTSS
CAHONS
CB2SFV
CB3-3V    Comments:
CBSFXS
CDM10E
CDM10G
CDM10I
CDM10P
CDM10S
CDM10V
CDM11E
CDM11G
                                                                   CL NL SL J1
5=Lookup,Copy,Print  6=Toggle     F10=More    Shft-Fn=Fn Help
```

SDG, Screen 4

4.6.3 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.

4.6.4 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.)

4.6.5 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.

4.6.6 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.

4.6.7 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.

4.7 Sample storage

4.7.1 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

4.7.2 A rush backlog report is printed throughout the day for short holding-time tests, with the exception of microbiology, in order that they be recognized by the analysts. **A checklist (Appendix 8) for analyses with short holding times is completed prior to release of samples to the laboratory. Sample management will verify that the correct RUSH test codes are entered, date, time received and collected dates are accurate and to ensure matrices and sample I.D.s are correct at time of log-in.**

4.7.3 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.

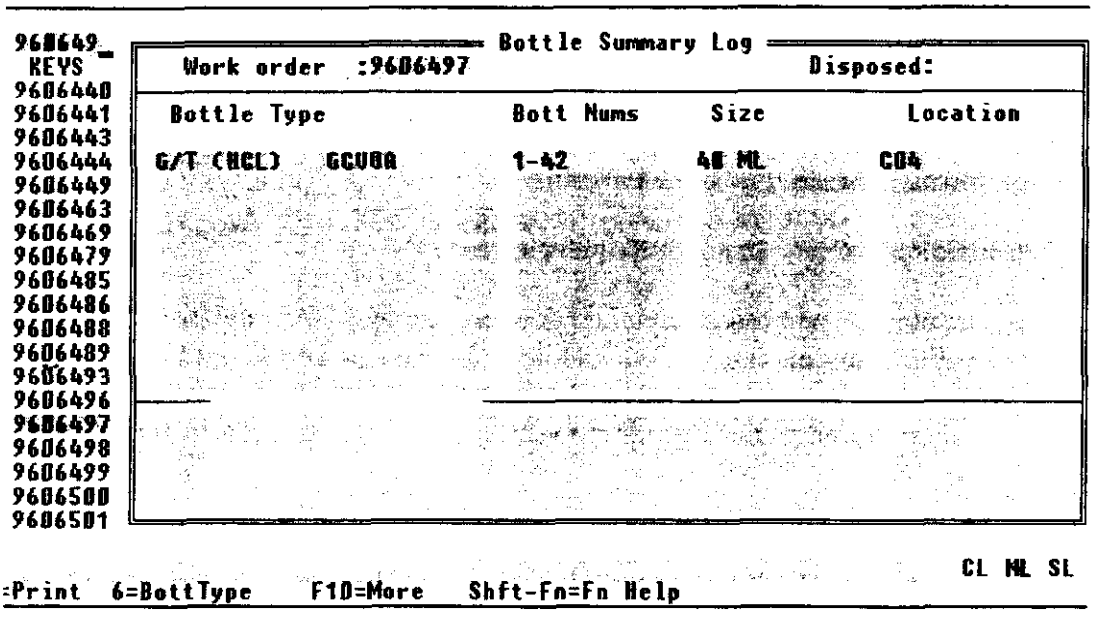
4.8 Storage locations

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.

4.8.1 *See Section 4.7 for transfer of extractable aliquots to the extractions lab

4.8.2 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.

4.8.3 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.



4.9 Determination of tests

4.9.1 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.

4.9.2 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.

4.9.3 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:

4.9.4 If volatiles are requested, then the VOA departments will get the samples first (GC or GC/MS)

4.9.5 The sample/samples will then be sent to the extractions lab

4.9.6 The extractions lab will return the sample/samples to the inorganics lab or other areas

4.9.7 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.

4.10 Electronic Transmittal of Sample and Test Request Records

4.10.1 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.

4.10.2 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.

4.10.3 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.

4.10.4 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.

4.10.5 The Project Manager makes corrections to these items as necessary, usually in consultation with the Sample Custodian. When transmittal is complete, the hard-copy record generated in the transmittal process is stapled to the supporting documentation previously assembled by the Sample Custodian and the complete record is filed alphabetically (by client name) in the filing drawer designated. If a CLP-style package is being generated, packets are prepared for the CLP Document Control Custodian.

4.10.6 Specific test requests are made known to analysts through hard-copy work "backlogs". For a description of this process, see the SOP on Data Handling.

4.11 Generation of internal Chain-of-Custody (COC)

4.11.1 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.

4.11.2 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.

4.11.3 The COC is returned to 940 with the extracts when extractions are completed.

4.12 Sample breakage

4.12.1 All sample breakage, whether in shipping or while handling in the lab, must be reported immediately to the Project Manager.

4.12.2 If the sample was water, clean thoroughly with disposable towels. Be very careful with broken glass so as to avoid cuts.

4.12.3 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.

4.12.4 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.

4.13 Special circumstances

4.13.1 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).

4.13.2 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

NON-CLP Sample Receipt Log

Laucks Testing Laboratories, Inc.
NON-CLP SAMPLE RECEIPT LOG

Initial once samples are checked in_____

DATE RECEIVED:_____ SAMPLE LOG-IN DATE:_____
TIME RECEIVED:_____ WORKORDER #:_____
CLIENT NAME:_____ CLIENT PROJECT:_____
SDG #_____ AIRBILL ATTACHED?:(#)_____
COC #_____ RECEIVED BY:_____

Non-Conformance: (Check applicable item(s)) Client IDs affected:

°(1) Not enough sample sent for proper analysis. #s affected:_____
□(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 3

CLP Sample Receipt Log

Supplemental Sample Receipt Log

COOLER RECEIPT FORM

WORKORDER #: _____

Contractor Cooler _____
QA Lab Cooler # _____
Number of Coolers _____

Project: _____

Date samples were received at the laboratory: ___/___/___

A. PRELIMINARY EXAMINATION PHASE: Date cooler was opened: ___/___/___

By: (print)_____(sign)_____

1. Did cooler come with a shipping slip (airbill, etc.),>>>>>>>>>>>>>>>>>>>>> YES NO
   If YES, record carrier name and airbill number: _____
2. Were custody seals on outside of cooler?>>>>>>>>>>>>>>>>>>>>>>>>>>>>> YES NO
   How many and where: _____
   Seal date: ___/___/___Seal name: _____
3. Were custody seals unbroken and intact at the date and time of arrival?>>>>>> YES NO
4. Did you screen samples for radioactivity using the Geiger Counter?>>>>>>>> YES NO
5. Were custody papers sealed in a plastic bag and taped inside to the lid?>>>>>> YES NO
6. Were custody papers filled out properly (ink, signed, etc.)?>>>>>>>>>>>>>>> YES NO
7. Did you sign custody papers in the appropriate place?>>>>>>>>>>>>>>>>>>>YES NO
8. Was project identifiable from custody papers? If YES, enter project name at top of this form.
9. If required, was enough cooling material present?>>>>Type of ice: _____YES NO
10. Have designated person initial here to acknowledge receipt of cooler: ____date___/___/___

B. LOG-IN PHASE : Date samples were logged-in: ___/___/___

By (print) _____(sign)_____

11. Describe type of packing in cooler: _____
12. Were all bottles sealed in separate plastic bags?>>>>>>>>>>>>>>>>>>>>>>> YES NO
13. Did all bottles arrive unbroken and were labels in good condition?>>>>>>>> YES NO

Page 1 of 3

LAUCKS TESTING LABORATORIES, INC.
COOLER RECEIPT FORM (continued)

14. Were all bottle labels complete (ID, date, time signature, preservative, etc.)? YES NO
15. Did all bottle labels agree with custody papers?>>>>>>>>>>>>>>>>>>>>>>>YES NO
16. Were correct containers used for the tests indicated?>>>>>>>>>>>>>>>>>>>YES NO
17. Were correct preservatives added to samples?>>>>>>>>>>>>>>>>>>>>>>>>>YES NO
18. Was a sufficient amount of sample sent for tests indicated?>>>>>>>>>>>>>> YES NO
19. Were bubbles absent in VOA samples: If NO, list by QA #:>>>>>>>>>>>>> YES NO
20. Was project manager called / faxed & status discussed? >>>>>>>>>>>>>>>> YES NO
    If YES, give details below
21. Who was called / faxed? _____
    By whom?_____(date)_____

DISCREPANCIES:

_____
_____
_____
_____
_____
_____
_____
_____
_____
_____
_____
_____
_____
_____
_____
_____
_____
_____
_____
_____

Page 2 of 3

Laucks Testing Laboratories, Inc., Supplemental Sample Receipt Log

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

Page 3 of 3

APPENDIX 4

Secure Storage Custody Log

Organic Extractions Custody Log

Laucks Testing Laboratories, Inc.

Secure Storage Custody Log

Project:_____ **LTL Number:**_____

Number of Containers (*optional*):_____

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

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

Samples Disposed of by _____ on _____

Laucks Testing Laboratories, Inc.

Organic Extractions Custondy Log

Samples Entered By	Time	Date	JOB #	
			Client	
Samples Moved From Bldg. 940 to 921 by:			*Matrix*	*Sample #*
			SOIL	
			WATER	
			SLUDGE	
			MISC.	
			OIL	

Comments:

APPENDIX 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 #	Sample #	WE HAVE	WE NEED	TOTAL	DISS	UNPRES	INTL & DATE	BOTTLE TYPES
								Y = YELLOW
								B = BLUE
								C = CYANIDE
								R = RED
								O = O&G
								P = PHENOL
								S = SULFIDE
								T = TOC
								OTHER

APPENDIX 7

Required Containers/Volumes/Preservation/Holding Times

REQUIRED CONTAINERS/VOLUMES, PRESERVATION TECHNIQUES AND MAXIMUM HOLDING TIMES FOR ENVIRONMENTAL ANALYSIS

A. PRIORITY POLLUTANTS - Organics Analysis (Federal Register Vol. 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

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	

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

B. WASTE EVALUATION - ORGANICS ANALYSIS (SW-846, 3rd Edition) (continued)

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Volatile Organics (VOAs)	2- 40 ml containers for liquid sample 20 grams for solids	Glass, Teflon-lined Septum 40 ml capacity As above or glass, 2-4 oz.	Cool, 4° C, acid preserved with HCl to pH < 2, no headspace Cool, 4° C, no headspace or if solid packed to minimize headspace	14 days 14 days	Method 8260 Purge-and-Trap GC/MS
Semi-Volatile Organics	Approximately 1 liter for liquid sample Approximately 50 grams for sludge or solid sample	Glass, Teflon-lined cap	Cool, 4° C,	Extracted within 7 days and completely analyzed within 40 days Extracted within 14 days and completely analyzed within 40 days	Method 8270 GC/MS

C. WASTE EVALUATION - GENERAL

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Toxicity Characteristic Leaching Procedure	Approximately 1 liter for liquid sample *Approximately 200 grams* for solid sample	Glass, Teflon-lined cap	VOA, Metals, Semivolatiles, Pesticides/Herbicides - 14 days until extraction. Follow analytical protocol for aqueous holding time or holding time from leachate preparation.	Not specified	According to requested analysis
Water Reactivity	Approximately 100 ml for liquid sample *Approximately 50 grams* for solid sample	-----	None	14 day	-----

C. WASTE EVALUATION - GENERAL (continued)

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Ignitability	Approximately 200 ml or 50 grams	None specified	None	14 days	Method 1010 Pensky-Martens Closed-Cup Method Method 1020 Setaflash Closed Cup Method
Corrosivity	100 - 500 ml	Plastic	None	None	Method 1110 Corrosivity Toward Steel
California Assessment Manual CAM/STLC	Approximately 200 ml for liquid sample Approximately 10 grams for solid sample	Plastic or Glass	No preservation for solid sample. Add HNO_3 to pH <2 for liquid sample	28 days for Mercury 6 months for others	See Methods for Metals Analysis

D. METALS ANALYSIS (EPA Methods for Chemical Analysis of Water and Wastes, March 1983, or APHA Standard Methods, 15 Edition. and EPA SW-846, 3rd Edition)

PARAMETER	MINIMUM VOLUME REQUIRED **	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Mercury, Total/Dissolved	100-200 ml for liquid sample	Plastic or Glass	HNO_3 to pH <2 for total	28 days	EPA 245.1 for water or EPA 7470A
Mercury, Total	Approximately 5 grams for solid sample		Filter on site, HNO_3 to pH <2 for dissolved		EPA 7471A for sediment Cold Vapor Method
Metals, Total Metals, Dissolved	300 ml for liquid samples Approximately 10 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 for total Filter on site, HNO_3 to pH <2 for dissolved	6 months	Flame AA-- See Individual Metal Methods Emission AA-- See Individual Metal Methods Graphite Furnace AA- See individual Metal Methods or ICP-- 200.7 or 6010

**** For individual metals the aggregate minimum volume is determined by the number of discrete analytical methods not the sum of all the individual analytes.**

D. METALS ANALYSIS (EPA Methods for Chemical Analysis of Water and Wastes, March 1983, or APHA Standard Methods, 15 Edition. and EPA SW-846, 3rd Edition) (continued)

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Hexavalent Chromium -Cr^6	200 ml for liquid sample Approximately 50 grams for solid samples	Plastic or Glass	Cool, 4 C	24 hours Extracted within 7 days, analyzed within 24 hrs. of extraction.	EPA 218.4 or EPA 7196A EPA 218.5 or EPA 7197 Extraction/AA Method EPA 7196 Colorimetric Method
Aluminum (Al)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 202.1, Flame EPA 202.2 or EPA 7020, Furnace
Antimony (Sb)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 204.1 or EPA 7040, Flame EPA 204.2 or EPA 7041, Furnace
Arsenic (As)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 206.3 or EPA 7061, Hydride AA EPA 206.2 or EPA 7060, Furnace AA
Barium (Ba)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 208.1 or EPA 7090, Flame AA EPA 208.2, Furnace AA
Beryllium (Be)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 210.1 or EPA 7090, Flame AA EPA 210.2A or EPA 7091, Furnace AA
Boron (B)	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic only	Cool, 4° C	6 months	Curcumin Colorimetric EPA 212.3

**** Each metal can also be analyzed by EPA 200.7, EPA 6010A, or EPA 6020.**

D. METALS ANALYSIS (EPA Methods for Chemical Analysis of Water and Wastes, March 1983, or APHA Standard Methods, 15 Edition. and EPA SW-846, 3rd Edition) (continued)

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Cadmium (Cd)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 213.1 or EPA 7130, Flame AA EPA 213.2 for EPA 7131, Furnace AA
Calcium (Ca)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 215.1 or EPA 7140, Flame AA
Chromium (Cr)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 218.1 or EPA 7190, Flame AA EPA 218.2 or EPA 7191, Furnace AA EPA 218.3 or EPA 7198, Chelation Extraction
Cobalt (Co)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 219.1 or EPA 7200, Flame AA EPA 219.2 or EPA 7201, Furnace AA
Copper (Cu)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 220.1 of EPA 7210, Flame AA EPA 220.2, Furnace AA
Gold (Au)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 231.1, Flame AA EPA 231.2, Furnace AA
Iron (Fe)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 236.1 or EPA 7380, Flame AA EPA 236.2, Furnace AA

**** Each metal can also be analyzed by EPA 200.7, EPA 6010A, or EPA 6020.**

D. METALS ANALYSIS (EPA Methods for Chemical Analysis of Water and Wastes, March 1983, or APHA Standard Methods, 15 Edition. and EPA SW-846, 3rd Edition) (continued)

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Lead (Pb)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 239.1 or EPA 7420, Flame AA EPA 239.2 or EPA 7421, Furnace AA
Magnesium (Mg)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 242.1 or EPA 7460, Flame AA
Manganese (Mn)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 243.1 or EPA 7480, Flame AA EPA 243.2 or EPA 7481, Furnace AA
Molybdenum (Mo)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 246.1 or EPA 7520, Flame AA EPA 246.2, Furnace AA
Nickel (Ni)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 249.1 or EPA 7610, Flame AA EPA 249.2, Furnace AA
Potassium (K)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 258.1, Flame AA
Selenium (Se)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 270.2 or EPA 7740, Furnace AA EPA 270.3 or EPA 7741, Hydride AA

**** Each metal can also be analyzed by EPA 200.7, EPA 6010A, or EPA 6020.**

D. METALS ANALYSIS (EPA Methods for Chemical Analysis of Water and Wastes, March 1983, or APHA Standard Methods, 15 Edition. and EPA SW-846, 3rd Edition) (continued)

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Silver (Ag)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 272.1 or EPA 7760, Flame AA EPA 272.2, Furnace AA
Sodium (Na)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 273.1 or EPA 7770, Flame AA
Thallium (Tl)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C.	6 months	EPA 279.1 or EPA 7840, Flame AA EPA 279.2 of EPA 7841, Furnace AA
Tin (Sn)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 282.1 or EPA 7870, Flame AA EPA 282.2, Furnace AA
Titanium (Ti)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 283.1, Flame AA EPA 283.2, Furnace AA
Vanadium (V)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 286.1 or EPA 7910, Flame AA EPA 286.2 or EPA 7911, Furnace AA
Zinc (Zn)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 289.1 or EPA 7950, Flame AA EPA 289.2, Furnace AA

**** Each metal can also be analyzed by EPA 200.7, EPA 6010A, or EPA 6020.**

E. GENERAL: MINERAL ANALYSIS/VOLATILES - DRINKING WATER or TITLE 22 CAL/DOHS

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Complete General Mineral Analysis	1 - 2 liters	Plastic or Glass	Cool, 4° C, additional preservation depends on the analyte list	-----	-----
pH	50 ml	Plastic or Glass	None	Immediate	EPA Method 150.1 pH Meter
Alkalinity	50 - 100 ml	Plastic or Glass	Cool, 4° C	14 days	EPA Method 310.1 Titrimetric Method
Calcium**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	Flame AA EPA 215.1
Chloride	50 - 100 ml	Plastic or Glass	Cool, 4° C	28 days	EPA Method 325.3 Titrimetric Method
Copper**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	Flame AA EPA 220.1
MBAS	500 ml	Plastic or Glass	Cool, 4° C	48 hours	EPA Method 425.1 Colorimetric
Iron**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	Flame AA EPA 236.1
Magnesium**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	Flame AA EPA 242.1
Manganese**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	Flame AA EPA 243.1
Sodium**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	Flame AA EPA 273.1
Sulfate	50 - 100 ml	Plastic or Glass	Cool, 4° C	28 days	EPA 375.4 Turbidimetric
Electrical Conductivity	50 - 100 ml	Plastic or Glass	Cool, 4° C	24 hrs. or filter	EPA Method 120.1 EC Water
Total Dissolved Solids	100 ml	Plastic or Glass	Cool, 4° C	7 days	EPA Method 160.1 Gravimetric
Total Hardness	50 - 100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	EPA Method 130.2 Titrimetric Standard Method 314-A Calculation
Zinc**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	Flame AA EPA 289.1
Volatile Organics (VOAs)	2 - 40 ml containers	Glass, Cap teflon-lined, 40 ml.	No headspace, HCl to pH<2, if residual chlorine then preserve with Na_2SO_4 or Ascorbic Acid Wash. State - No headspace and HCl to pH <2 only	14 days	EPA Method 524.2

**** Each metal can also be analyzed by EPA 200.7 or EPA 6010A.**

F. INORGRANIC ANALYSIS: DRINKING WATER or TITLE 22 - CAL/DOHS

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Arsenic**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	EPA 206.3, Furnace AA
Barium**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	EPA 208.1, Flame AA
Cadmium**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	EPA 213.1 , Flame AA or EPA 218.2, Furnace AA
Chromium**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	EPA 218.1, Flame AA EPA 218.2, Furnace AA
Lead**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	EPA 239.2, Furnace AA
Mercury	100 ml	Plastic or Glass	HNO_3 to pH <2	28 days	EPA 245.1 ,Cold Vapor
Selenium**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	EPA 270.2 ,Furnace AA
Silver**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	EPA 272.1 ,Flame AA
Nitrate-Nitrogen	50 ml	Plastic or Glass	Cool, 4° C add H_2SO_4 to pH <2	14 days	EPA 352.1, Brucine Sulfate
			Cool, 4° C	48 hours	EPA 353.3, Cadmium Reduction
Fluoride	300 ml	Plastic or Glass	None	28 days	EPA 340.2, Ion Selective Electrode

**** Each metal can also be analyzed by EPA 200.7 or EPA 200.8.**

G. GENERAL PHYSICAL ANALYSIS: DRINKING WATER or TITLE 22 - CAL/DOHS

PARAMETER	VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Color	50 ml	Plastic or Glass	Cool, 4° C	48 hours	EPA Method 110.2
Odor	200 ml	Plastic or Glass	Cool, 4° C	48 hours	EPA Method 180.1, Threshold Odor
Turbidity	100 ml	Plastic or Glass	Cool, 4° C	48 hours	EPA Method 180.1, Nephelometric

H. GENERAL WET CHEMISTRY AND MISCELLANEOUS ANALYSIS

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Acidity	100 ml	Plastic or Glass	Cool, 4° C	14 days	EPA 305.1, Titrimetric
Alkalinity	50-100 ml	Plastic or Glass	Cool, 4° C	14 days	EPA 310.1 ,Titrimetric
Ammonia Nitrogen	100 ml	Plastic or Glass	Cool, 4° C H_2SO_4 to pH <2	28 days	EPA 350.1, Colorimetric
BOD	1 liter	Plastic or Glass	Cool, 4° C	48 hours	EPA 405.1
Boron	100 ml	Plastic	None	28 days	EPA 212.3 ,Curcumin
Chloride	100 ml	Plastic or Glass	None	28 days	EPA Method 325.3, Mercuric Nitrate or EPA 300.0, Ion Chromatography
COD	20 ml	Plastic or Glass	Cool, 4° C H_2SO_4 to pH <2	28 days	EPA Methods 410.4, Colorimetric
Coliform Fecal Coliform	100 ml	Sterilized Plastic Bottles	Cool, 4° C $Na_2S_2O_3$ preserved for presence of free chlorine	6 hours or 30 hours depending on the test requested	Standard Method 909A or 909C
Color	50 ml	Plastic or Glass	Cool, 4° C	48 hours	EPA 110.2 or 110.3
Cyanide	500 ml	Plastic or Glass	Cool, 4° C NaOH to pH >12	14 days	EPA 335.3, Colorimetric
Electrical Conductivity	50 - 100 ml	Plastic or Glass	Cool, 4° C	28 days	EPA 120.1, EC Meter
Fluorides	300 ml	Plastic or Glass	None	28 days	EPA 340.2, Ion Specific Electrode or EPA 300.0, Ion Chromatography
MBAS	500 ml	Plastic or Glass	Cool, 4° C	48 hours	EPA 425.1, Colorimetric

H. GENERAL WET CHEMISTRY AND MISCELLANEOUS ANALYSIS (continued)

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Nitrate Nitrogen	100 ml	Plastic or Glass	Cool, 4° C	14 days	EPA 300.0, Ion Chromatography
Nitrate Nitrite Nitrogen	100 ml	Plastic or Glass	Cool, 4° C Add H_2SO_4 to pH <2	14 days	EPA 353.3, Cadmium Reduction
Nitrite Nitrogen	50 ml	Plastic or Glass	Cool, 4° C	48 hours	EPA 354.1, SPectrophotometric
Odor	200 ml	Plastic or Glass	Cool, 4° C	24 hours	EPA 140.1
Oil and Grease	1 liter	Glass	Cool, 4° C Add H_2SO_4 to pH <2	28 days	EPA 413.1, Gravimetric
Orthophosphate	50 ml	Plastic or Glass	Cool, 4° C	48 hours	EPA 365.2, Ascorbic Acid
pH	50 ml	Plastic or Glass	None	Immediate	EPA Method 150.1, pH Meter
Phenolics	500 ml	Glass	Cool, 4° C Add H_2SO_4 to pH <2	28 days	EPA 420.1, 4-AAP
Radioactivity	1-18 liters	Plastic or Glass Glass only for Tritium	HCl or HNO_3 * to pH <2 *Some exceptions	-----	Standard Method 701
Silica	50 ml	Plastic	Cool, 4° C	28 days	EPA 370.1, Colorimetric Flame AA Method
Sulfates	50 ml - 100 ml	Plastic or Glass	Cool, 4° C	28 days	EPA 375.4, Turbidimetric or EPA 300.0, Ion Chromatography
Sulfides	500 ml	Plastic or Glass	NaOH to pH >9 2 ml Zinc Acetate Cool, 4° C	7 days	EPA 376.1 ,Titrimetric
Sulfites	50 ml	Plastic or Glass	None	Immediate	EPA 377.1 ,Titrimetric
TOC	25 ml	Plastic or Glass	Cool, 4° C H_2SO_4 to pH <2	28 days	EPA 415.2, TOC Analyzer
Total Dissolved Solids	100 ml	Plastic or Glass	Cool, 4° C	7 days	EPA 160.1, Gravimetric

H. GENERAL WET CHEMISTRY AND MISCELLANEOUS ANALYSIS (continued)

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Total Hardness	50-100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	EPA 130.2, Titrimetric Standard Method 314-A Calculation
Total Kjeldahl Nitrogen	1 liter	Plastic or Glass	Cool, 4° C Add H_2SO_4 to pH <2	28 days	EPA 351.4, Ion Specific Electrode
Total Organic Nitrogen TON-TKN-NH_3-N	1 liter	Plastic or Glass	Cool, 4° C Add H_2SO_4 to pH <2	28 days	EPA 351.4 and EPA 350.3
Total Petroleum Hydrocarbons Scan, by GC	400 ml to 1 liter Approximately 50 grams for a solid sample	Glass	Cool, 4^0 C	14 days, but this may vary between states so regulations must be consulted	WTPH-HCID by GC/FID or Modified Method 8015, GC
Total Petroleum Hydrocarbons as Gas, by GC	40 ml Approximately 20 grams for a solid sample	Glass	Cool, 4^0 C, Methanol preservation may be a requirement of some states so state regulations must be consulted Cool, 4^0 C	14 days	WTPH-G by purge and trap GC/FID or Modified Method 8015, GC/FID
Total Petroleum Hydrocarbons as Diesel, by GC	1 liter Approximately 50 grams for a solid sample	Glass	Cool, 4^0 C	14 days	WTPH-D by GC/FID or Modified Method 8015, GC/FID
Total Phosphate	50 ml	Plastic or Glass	Cool, 4° C Add H_2SO_4 to pH <2	28 days	EPA 365.4, Colorimetric
Total Residue	100 ml	Plastic or Glass	Cool, 4° C	7 days	EPA 160.3 , Gravimetric
Total Settleable Solids	1 liter	Plastic or Glass	Cool, 4° C	48 hours	EPA 160.5, Imhoff Cone
Total Suspended Solids	100 ml	Plastic or Glass	Cool, 4° C	7 days	EPA 160.2, Gravimetric
Total Volatile Solids	100 ml	Plastic or Glass	Cool, 4° C Add H_2SO_4 to pH <2	7 days	EPA 160.4, Gravimetric
TOX	500 ml	Amber Glass, Teflon Septum	Cool, 4° C Add H_2SO_4 to pH <2	7 days	EPA 450.1 or EPA 9020, TOX Analyzer
Turbidity	100 ml	Plastic or Glass	Cool, 4° C	48 hours	EPA 180.1

APPENDIX 8

Checklist For Analyses With Short Holding Times

CHECKLIST FOR ANALYSES WITH SHORT HOLDING TIMES

To be completed prior to release of release of samples to laboratory

_____ Correct test codes are entered

_____ Dates and times received and collected are correct

_____ Matrices are correct

_____ Sample I.D.s are correct

I certify that all of the above have been checked and were found accurately entered in the LIMS.

_____ _____
(Signature) (Date)

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-7202

Title: Metals Analysis Using Inductively Coupled Plasma - Mass Spectrometry (ICP/MS), SW 846 Method 6020

Revision history:

Number	Date
1.0	04/26/95
2.0	02/09/98
3.0	4/22/98
4.0	7/22/99

Written by: _____ Date: 7-22-99
Ming-Hwa Hwang, Metals Supervisor

Approved by: _____ Date: 7-22-99
Harry Romberg, QA Officer

Approved by: _____ Date: 7-22-99
Kathy Kreps, Laboratory Director

Controlled Document
No. 20 Assigned to: Tetra

Table of Contents

1. INTRODUCTION AND SCOPE 4

1.1 METHOD DESCRIPTION 4
1.2 SAMPLE COLLECTION, SAMPLE STORAGE, HOLDING TIMES 4
1.3 DEFINITION OF TERMS 4

2. EQUIPMENT LIST AND STANDARDS 7

2.1 INSTRUMENTATION: 7
2.2 STANDARDS 7
2.3 INTERNAL STANDARDS 7

3. SAFETY PRECAUTIONS AND WASTE DISPOSAL 7

3.1 SAFETY PRECAUTIONS 7
3.2 WASTE DISPOSAL 8

4. QUALITY CONTROL 8

4.1 METHOD DETECTION LIMIT STUDY 8
4.2 LINEAR RANGE STUDY 8
4.3 INTERNAL STANDARDS 8
4.4 INITIAL CALIBRATION 9
4.5 INITIAL CALIBRATION VERIFICATION 9
4.6 INITIAL CALIBRATION BLANK 10
4.7 CONTINUING CALIBRATION VERIFICATION (CCV) AND BLANK (CCB) 10
4.8 INTERFERENCE CHECK SOLUTIONS A (ICSA) AND AB (ICSAB) 10
4.9 METHOD BLANKS 11
4.10 LABORATORY CONTROL SAMPLE 12
4.11 MATRIX SPIKE 12
4.12 POST-DIGESTION SPIKE 13
4.13 MATRIX SPIKE DUPLICATE/SAMPLE DUPLICATE 13
4.14 SERIAL DILUTION 14

5. PROCEDURE 15

5.1 INSTRUMENTAL CONDITIONS 15
5.2 ANALYTICAL OPERATION 15

6. REPORTS 16

6.1 DATA PACKET ORGANIZATION 16
6.2 REFERENCES: 16

APPENDIX I 17

STANDARD SOLUTIONS 17

APPENDIX II 20

ICSA AND ICSAB SOLUTIONS 20

APPENDIX III 22

QC SUMMARY TABLE 22

APPENDIX IV **25**

FLOW CHART 25

APPENDIX V **27**

ROUTINE REPORTING LIMITS 27

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 sample should be recovered to within 75% to 125% of the known value or within the laboratory derived acceptance criteria.

Standard-Addition - The standard addition technique involves adding known amounts of standard to an aliquot of the sample. This technique compensates for a sample constituent that enhances or depresses the analyte signal thus producing a different slope than that of the calibration standards.

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

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

2. Equipment List and Standards

2.1 Instrumentation:

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 20 ppm of Sc45, 10 ppm of In115 and 10 ppm of Bi209 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 interfering 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 oncentration of the blank of any analyte of concern should not be higher than the highest of ither:

(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 the sample result is greater than 4 times the spike concentration or unless otherwise specified in the project specific QAPP. In-house control limits are based on historical performance.
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 original 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 concentrations greater than 5 times the CRDL, control limits for RPD of duplicates will be ±20% unless otherwise specified in the project specific QAPP. For sample concentrations less than 5 times the CRDL, control limits for the difference will be ± the CRDL. In-house control limits are based on historical performance. The RPD control limits are detailed in the current QC Database 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 50 times greater than the IDL, then results for those analytes will be flagged with an "E".

Corrective Action:

No corrective is necessary other than appropriately flagging the data.

5. Procedure

5.1 Instrumental Conditions

- Refer to manufacture's instruction for specific operating procedures. Allow at least 30 minutes for the instrument to stabilize before initiating any analysis.

- Conduct mass calibration and resolution checks in the mass regions of interest. The mass calibration and resolution parameters are required criteria which must be met prior to any sample being analyzed. The monitored masses of Mg, Rh, and Pb must meet the following criteria:

Element	RSD for replicate (minimum of four) integrations.	Mass, amu	Resolution @ 10 % peak height, amu
Mg	< 5%	23.90-24.10	<0.9
Rh	< 5%	102.80-103.00	<0.9
Pb	< 5%	207.90-208.10	<0.9

5.2 Analytical Operation

- Calibrate the instrument, using a calibration blank and a standard. Refer to Appendix I for the applied levels of concentration.

- All masses which could affect data quality are monitored to determine potential effects from matrix components on the analytes of interest.

- After the calibration has been established, an ICV solution is analyzed to verify the validity of the curve. Measurements for the analytes of interest must be at ±10% of the true value. A re-calibration and re-analysis is required for any analyte which falls outside the control limit.

- Analyze the interference check samples (ICSA and ICSAB) prior to and after the analysis of samples

- Analyze a CCV and a CCB once every 10 analytical samples.

- Dilute samples that exceed the established linear range of the instrument.

6. Reports

6.1 Data Packet Organization

- See the SOP metals validation for a check list detailing data packet organization
- If requested, all analysis performed under SW 846 guidelines the data can be reported via CLP SOW 3/90 forms.
- Data packages will be produced via Enviroforms. Analyte levels that are less than the MDL will be reported as the SDL followed by a "U". Analyte levels that fall between the MDL and the reporting limit will be flagged with a "B". Analyte levels greater than or equal to the reporting limit PQL will be reported without a flag.

CODE	Definition	
U	The analyte of interest was not detected, to the limit of detection indicated.	✱◆
B	The analyte of interest was detected between the MDL and the reporting limit.	✱
N	The spike recovery exceeded the control limits.	✱
*	The duplicates exceeded the RPD control limit or their difference exceeded the reporting limit.	✱
E	The Serial Dilution did not agree within 10%.	✱
S	The analyte concentration was determined by MSA.	✱

◆ Used in all reports.
✱ Used in data validatable packages.

6.2 References:

Test Methods for Evaluating Solid Waste, SW-846, Method 6020, Revision 0, September 1994

Appendix I

Standard Solutions

Elements	Mass	STD, µg/L
Be	9	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.

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.

Appendix II

ICSA and ICSAB Solutions

Element	ICSA (PPM)	ICSAB (PPM)
Aluminum	4	4
Arsenic		0.020
Cadmium		0.020
Calcium	12	12
Chromium		0.040
Cobalt		0.040
Copper		0.040
Iron	10	10
Magnesium	4	4
Manganese		0.040
Molybdenum		0.16
Nickel		0.040
Potassium	4	4
Selenium		0.020
Silver		0.040
Sodium	10	10
Vanadium		0.040
Zinc		0.020

Appendix III

QC Summary Table

Laucks Testing Laboratories

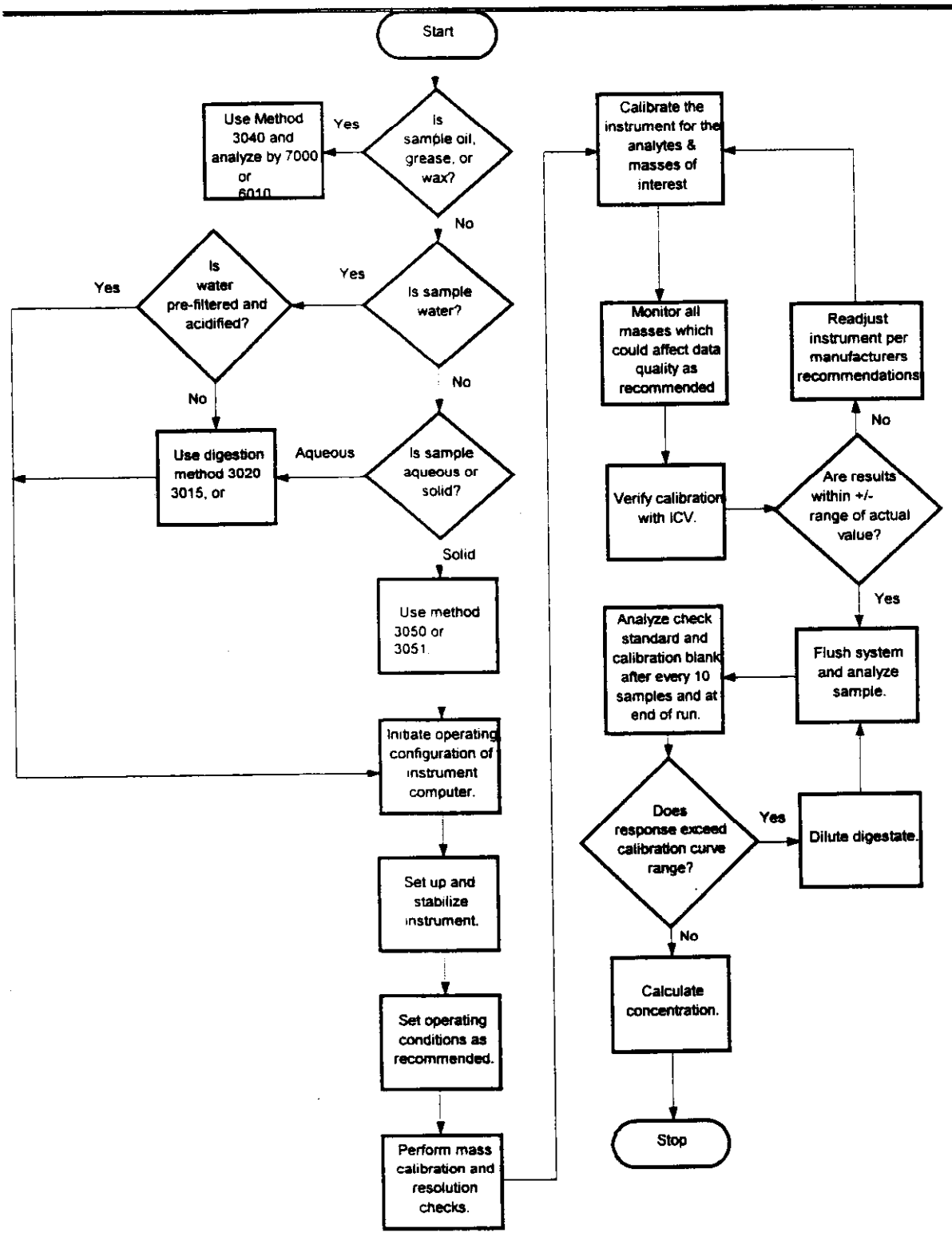
Method SW 846 6020 QA Requirements and Corrective Actions

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 independent source.	±10% of true value. Made from an independent source..	Immediately following calibration.	Recalibrate and reverify.	Form 2, in the raw data
Initial Calibration Blank	Values must be < 3x the IDL for each element.	Values must be < 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, % Difference	± 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
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
Ag	3.0	Mn	1.0
As	1.0	Mo	10.0
B	10.0	Ni	10.0
Ba	2.0	Pb	1.0
Be	1.0	Sb	1.0
Cd	1.0	Se	3.0
Co	3.0	Sn	10.0
Cr	5.0	U	100
Cu	2.0	V	2.0
Li	10.0	Zn	10.0

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-8081

Title: **Analysis of Organochlorine Pesticides and PCBs by Method SW 8081**

Revision history:

Number	Date
1.0	9/25/89
2.0	2/3/93
3.0	12/22/95
4.0	8/11/97
5.0	2/2/98
6.0	7/10/98

Revised by: Monica Carr, Organic Division Manager Date: 7/10/98

Approved by: Kathy Kreps, Laboratory Director Date: 7-10-98

Approved by: Harry Romberg, QA Officer Date: 7-10-98

Controlled Document

No. 20 Assigned to: Tetra

TABLE OF CONTENTS

1. INTRODUCTION AND SCOPE 4

1.1 METHOD DESCRIPTION 4
1.2 SAMPLE COLLECTION, SAMPLE STORAGE, HOLDING TIMES 5
1.3 DEFINITION OF TERMS 5

2. EQUIPMENT LIST AND STANDARDS 8

2.1 CHROMATOGRAPHIC SYSTEM 8
2.2 STANDARDS 8

3. SAFETY PRECAUTIONS AND WASTE DISPOSAL 9

3.1 ROUTINE SAFETY PRECAUTIONS 9
3.2 WASTE DISPOSAL 10

4. CALIBRATION AND QUALITY CONTROL 10

4.1 QA REQUIREMENTS AND CORRECTIVE ACTION 10
4.2 METHOD DETECTION LIMIT STUDY 10
4.3 METHOD VALIDATION 10
4.4 RETENTION TIME WINDOWS 11
4.5 BREAKDOWN EVALUATION 12
4.6 INITIAL MULTI-POINT CALIBRATION 12
4.7 EXTERNAL STANDARD CALIBRATION 12
4.8 INITIAL CALIBRATION VERIFICATION 13
4.9 UPDATING RETENTION TIME WINDOWS 14
4.10 INSTRUMENT BLANK 14
4.11 METHOD DETECTION LIMIT STANDARD 15
4.12 CONTINUING CALIBRATION VERIFICATION 15
4.13 METHOD BLANKS 16
4.14 METHOD BLANK SPIKES 16
4.15 MATRIX SPIKE 17
4.16 MATRIX SPIKE DUPLICATE 17
4.17 SURROGATE RECOVERY 18

5. OPERATION PROCEDURES 19

5.1 CHROMATOGRAPHIC CONDITIONS 19
5.2 SAMPLE ANALYSIS 20

6. REPORTS 23

6.1 DATA PACKET ORGANIZATION 23
6.2 QUALITY CONTROL REPORTS 23
6.3 SAMPLE RESULT REPORTS 23
6.4 CONTROL CHART(S) 23

7. REFERENCES 24

APPENDIX I 25
STANDARD SOLUTION CONCENTRATIONS, µG/L 25
APPENDIX II 28
ANALYSIS SEQUENCE 28
APPENDIX III 29
DATA PACKET ORDER 29
APPENDIX IV 32
METHOD 8081 QA REQUIREMENTS AND CORRECTIVE ACTIONS: 32
APPENDIX V 33
COMPOUND ELUTION ORDER ON DB5 AND DB608 MEGABORE (0.45 OR 0.53 MM ID) COLUMNS 33
APPENDIX VI 34
COMPOUND ELUTION ORDER ON DBXLB AND DB17MS CAPILLARY (0.25 MM ID) COLUMNS 34
APPENDIX VII 35
FLOW CHART 35

1. Introduction and Scope

1.1 Method Description

1.1.1 This method describes the procedures and specifications for instrumental analysis of various organochlorine pesticides and polychlorinated biphenyls (PCBs) in water and soil following SW846 Method 8081. Analysis is performed by gas chromatography using a single injection port and splitting into dual GC columns with electron-capture detectors. This system provides quantitation and confirmation of pesticides and Aroclors (PCBs) from a single injection. The following table lists the compounds that may be determined by this method. Additional compounds, listed in Method 8081, may also be determined.

ROUTINE COMPOUNDS	
alpha-BHC	beta-BHC
delta-BHC	gamma-BHC (Lindane)
Heptachlor	Aldrin
Heptachlor epoxide	Endosulfan I
Dieldrin	4,4'-DDE
Endrin	Endosulfan II
4,4'-DDD	Endosulfan sulfate
4,4'-DDT	Methoxychlor
Endrin aldehyde	Endrin ketone
alpha-Chlordane	gamma-chlordane
Toxaphene	Aroclor-1016
Aroclor-1221	Aroclor-1232
Aroclor-1242	Aroclor-1248
Aroclor-1254	Aroclor-1260

OPTIONAL COMPOUNDS	
*Chlordane (not otherwise specified)	Isodrin
Simazine	Atrazine

*When chlordane is requested, a technical chlordane multicomponent standard is analyzed and response factors are calculated for 3-5 major peaks (including the alpha- and gamma-isomers). If these peaks are present in the sample at similar ratios to those found in the technical chlordane standard, an average concentration is calculated from the peaks chosen for calibration. If the peaks are present in the sample at dissimilar ratios to those found in the technical chlordane standard, a calibration factor will be calculated for the standard

based upon the sum of the responses for each of the peaks and the sample concentration will be calculated based on the sum of the same peaks in the sample.

1.1.2 Method Deviations: The analyte list is based on Method 8081, additional analytes are only added if requested by the client(s).

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

1.2 Sample Collection, Sample Storage, Holding Times

1.2.1 Samples are normally collected in glass containers with Teflon-lined caps. All samples and sample extracts are stored at 4°C±2°C. Water samples must be extracted within 7 days of sample collection, soil samples within 14 days of sample collection. All extracts must be analyzed with 40 days of sample preparation.

1.3 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.

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.
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 NFESC work. In the context of this SOP, a blank spike is the same as a QC check standard. See also QC check standard.

CCV	Continuing calibration verification. This is the same acronym used in the CLP program. This is a standard analyzed at some prescribed frequency during the analysis sequence to verify that the instrument has remained in calibration.
CF	Calibration factor. The ratio of analyte instrument response to nanograms injected. This term is defined in the same way in both the CLP contract and SW 846.
CLP	Contract Laboratory Program. The USEPA program that contracts with laboratories to provide laboratory services. The term has come to mean a much broader set of methods and deliverables. In the context of this SOP, CLP means procedures or operations which are detailed in the CLP contract and which are extended to a broader working definition.
DIW	Deionized water. Lab reagent water. Organic-free water. Since the systems used to provide DIW at Laucks all contain carbon polishing filters, they are capable of providing organic-free water for use in method blanks and method blank spikes.
IBLK	Instrument blank. This term is borrowed from CLP. Blank solvent containing the method surrogates is injected into the instrument to monitor for carry over between sample extract injections.
ICV	Initial calibration verification. It is a standard which is injected at the start of each QC period that is compared to the initial multi-point calibration to determine whether the instrument is still in calibration.
IDL	Instrument detection limit. The lowest concentration of a target analyte that will yield a signal:noise ratio of at least 3x. Used as a starting point for selecting MDL study spiking levels.
MDL	Method detection limit. The lowest concentration in a sample which will yield a positive result that is greater than zero at a known level of confidence. MDLs are empirically determined by Laucks.

MDL standard	Method detection limit standard. A standard prepared so that the concentrations of the target analytes are in the range of 1x to 4x the empirically determined MDLs on an extract/digest basis. This standard is used to verify that the instrument is capable of detecting the target analytes on an ongoing basis.
PQL or Reporting Limit	Practical Quantitation Limit or Reporting Limit- The value used when reporting a non-detect. It may be administratively, empirically or contractually set.
QC check standard	Quality control check standard. Referred to in this SOP as a blank spike. A QC check standard is a requirement of SW 846 method 8000 and is used to determine whether the analytical system is in control if MS/MSD recoveries are out of control. See also blank spike.
QC period	Quality control period. An analysis sequence initiated by the analysis of one or more standards, followed by sample extracts/digests, and terminated with a standard analysis. A QC period can be open-ended chronologically, but calibration verification must be documented using the procedures in this SOP.
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
RSD or %RSD	Relative standard deviation or percent relative standard deviation. The ratio of the standard deviation of a set of values to the mean of the set of values expressed as a percentage. A measure of the similarity of the values one to another.

RT, Retention time	The time (in minutes) at which a target analyte elutes from a chromatography column.
RT window	Retention time window. The +/- value which is applied to the ICV to establish the time range used to make tentative compound identifications.
Sequence	A set of sample extracts/digests and standard solutions introduced into an instrument in a chronologically continuous group. See also QC period.
SRM	Standard Reference Material - A material containing known quantities of target analytes in a homogeneous matrix which approximates the matrix of the samples being analyzed. It is used to establish that the analytical process is in control.

2. Equipment List and Standards

2.1 Chromatographic System

2.1.1 This analysis requires a gas chromatograph with a programmable oven, heated injection port, dual electron-capture detectors, autosampler and an electronic data-acquisition system.
Equipment list:
Gas chromatograph (Hewlett-Packard 5890 / 5890A)
Autosampler (Hewlett-Packard 7673A)
2 electron-capture detectors (Hewlett-Packard)
2 capillary chromatographic columns of dissimilar phase (J&W DB5 and DB608, 30m x 0.53mm megabore, J&W DB5 and DB608, 30m x 0.45mm megabore or J&W XLB and J&W17ms, 30m x 0.25mm capillary, or equivalents).
Helium carrier gas
5% methane/95% argon detector make-up gas

2.2 Standards

2.2.1 Commercially prepared, certified stock standards are used to prepare working solutions for all surrogates, calibration mixes, and spike mixes. The 2 calibration mixes (INDA and INDB) contain all of the individual pesticide analytes. The spike mix contains 6 representative individual pesticides. These are ordered from Restek, Supelco or an equivalent supplier. Stock standards must be replaced by the manufacturers expiration date.

2.2.2 Calibration standards are prepared at 5 different concentration levels by dilution of the stock standards with hexane. High level INDA and INDB mixtures are made at 16 times the PQL concentration. The lower levels are diluted by factors of 2, 4, 8 and 16. They must be replaced after 6 months or by the expiration date of the material they were made from, whichever is earlier.

2.2.3 Multicomponent analyte calibration standards (Aroclors, chlordane, toxaphene), with the exception of Aroclors 1016 and 1260, are prepared at one concentration level, by dilution of commercially-purchased stock solutions in hexane. Standards of the Aroclors 1016 and 1260 are combined in a mixture and prepared at five concentration levels. These must be replaced after 6 months or by the expiration date of the material they were made from, whichever is earlier.

2.2.4 A breakdown evaluation (EVAL) mix is prepared from dilution of stocks of 4,4'-DDT and endrin with hexane.

2.2.5 A surrogate mix is prepared from neat materials and contains 2,4,5,6-tetrachloro-meta-xylene (TCMX) and decachlorobiphenyl (DCB). These standards are made in hexane and added to all standard mixes (before dilution if applicable).

2.2.6 Refer to SOP LTL-1013 (Preparation, Storage, Shelf Life and Traceability Documentation of Standards and Reference Materials) for detailed instructions on standards preparation and storage.

2.2.7 Appendix I details the compounds and concentrations contained in all solutions.

3. Safety precautions and Waste Disposal

3.1 Routine Safety Precautions

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

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

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

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

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

3.1.6 The electron-capture detectors used in this analysis contain a radioactive source and should not be opened or otherwise tampered with.

3.2 Waste disposal

3.2.1 Solvent wastes generated in using this procedure (expired standards, old sample extracts, rinsates, etc.) should be emptied into the solvent waste container in the fume hood.

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

4. Calibration and Quality Control

4.1 QA Requirements and Corrective Action

4.1.1 Detailed in the following sections are applicable QA requirements and subsequent corrective actions to be applied to this analysis. A summary of these requirements can be found in Appendix IV.

4.2 Method Detection Limit Study

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

4.3 Method Validation

4.3.1 Prior to the analysis of any samples, it is necessary to validate the method. A method validation study is performed in a similar manner to an MDL study with the exception that a minimum of 4 replicates are required and the concentration levels are typically higher.

4.3.2 The precision of spike recoveries must meet or exceed the criteria tabulated in SW 846.

4.4 Retention Time Windows

4.4.1 Retention time window studies are conducted following the Laucks SOP on Establishing RTWs and the procedure detailed in SW 846, Method 8000B, which defines a window for each single and multi- component analyte based on the retention times taken from three standard injections over a 72 hour period.

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

4.4.3 The calculated retention time window half widths are typically unrealistic values. Therefore RT windows have been administratively set at the values listed in OLM03.1 for megabore analyses (see table below) and at ±0.03 for capillary analyses. These values are typically wider than 3 times the standard deviation as determined above, but are more realistic.

Analyte	Megabore	Capillary
alpha-BHC	±0.05	±0.03
beta-BHC	±0.05	±0.03
gamma-BHC (Lindane)	±0.05	±0.03
delta-BHC	±0.05	±0.03
Heptachlor	±0.05	±0.03
Aldrin	±0.05	±0.03
alpha-Chlordane	±0.07	±0.03
gamma-Chlordane	±0.07	±0.03
Heptachlor epoxide	±0.07	±0.03
Dieldrin	±0.07	±0.03
Endrin	±0.07	±0.03
Endrin aldehyde	±0.07	±0.03
Endrin ketone	±0.07	±0.03
4,4'-DDD	±0.07	±0.03
4,4'-DDE	±0.07	±0.03
4,4'-DDT	±0.07	±0.03
Endosulfan I	±0.07	±0.03
Endosulfan II	±0.07	±0.03
Endosulfan sulfate	±0.07	±0.03
Methoxychlor	±0.07	±0.03
Tetrachloro-m-xylene	±0.07	±0.03
Decachlorobiphenyl	±0.10	±0.03

Isodrin	±0.07	±0.03
Aroclors	±0.07	±0.03
Toxaphene	±0.07	±0.03
Chlordane	±0.07	±0.03

4.5 Breakdown Evaluation

4.5.1 At the beginning of each analysis sequence an evaluation (EVAL) mix must also be analyzed. This is a mid-level standard containing 4,4'-DDT and endrin, and is examined for the breakdown products of these analytes (4,4'-DDD, 4,4'-DDE, endrin aldehyde, endrin ketone) which indicate the need for GC system maintenance. The percent breakdown must be less than 15% for both Endrin and 4,4'-DDT.

4.6 Initial Multi-Point Calibration

4.6.1 Analyze single component pesticide standard solutions using at least 5 different concentration levels. The lowest concentration should be at a concentration near, but above, the method reporting limit or PQL. The highest concentration should define the upper usable working range of the detector. Inject the standard solutions from the lowest concentration to the highest.

4.6.2 Analyze multi-component pesticide and PCB standard solutions at one concentration level, with the exception of the Aroclor 1016/1260 mixed standard. This standard should be analyzed at 5 different concentration levels.

4.7 External Standard Calibration

4.7.1 External standard initial calibration data is evaluated by determining the %RSD of the calibration factors.

4.7.2 CFs are calculated using the equation:

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

4.7.3 The calculated CFs are tabulated and the %RSD calculated. There are no compound-specific criteria. All %RSDs must be within 20% for each analyte or averaged across all analytes in the calibration standard mix.

4.7.4 A set of three to five major peaks is selected for each multicomponent analyte. These should be characteristic of the multicomponent analyte in question. Retention time windows and calibration factors are generated for each of the peaks chosen.

4.7.5 Corrective action

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

4.8 Initial Calibration Verification

4.8.1 Concentration and/or CF Criteria

4.8.1.1 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.

4.8.1.2 Using the appropriate calculation technique (average CF) compute either CFs or concentration values. For linear calibrations the ICV standard can be verified by calculating either the percent difference of the percent drift.

4.8.1.2.1 The percent difference calculation compares the ICV CFs to the mean CFs from the initial multi-point calibration. The percent difference is calculated as follows:

$$\%D = \frac{Cv - \overline{CF}}{\overline{CF}} \times 100$$

where:

C_v = Calibration Factor

$\overline{CF}$ = Mean Calibration Factor

4.8.1.2.2 The percent drift calculation compares the ICV calculated concentrations to the theoretical (or actual) concentration of the ICV standard. The percent drift is calculated as follows:

$$\%D = \frac{C_c - C_T}{C_T} \times 100$$

where:

C_c = Calculated Concentration
C_T = Theoretical Concentration

4.8.1.3 There are no compound-specific criteria. The %D results should be within ±15% of the average CF or expected concentration from the initial calibration for each analyte, or averaged across all analytes in the continuing calibration standard.

4.8.2 Corrective action

4.8.2.1 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.

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

4.10.2 Corrective action

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

4.11.2 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 every 10 samples, or every 12 hours, whichever is sooner. In addition, this standard must be the last injection made in the analysis sequence.

4.12.2 Criteria

4.12.2.1 After every 10 sample extract injections, a CCV standard is analyzed. The CF or concentration for each analyte is calculated and the percent difference or percent drift is calculated as shown above.

4.12.2.2 The %D results should be within ±15% of the average CF or expected concentration from the initial calibration for each analyte, or averaged across all analytes in the continuing calibration standard.

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

4.12.3 Corrective action

4.12.3.1 Check calculations or perform instrument maintenance. To validate the quantitation of target analytes in analytical samples, the samples must be bracketed by in-control CCVs. However, CCV CFs can be outside the control limits as long as this was due to an increase in response and the corresponding samples contain no detectable levels of the target analyte for which the CF is out of control.

4.13 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 reporting limit is reported.

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 the sample group may not require re-extraction. In any event it is the laboratory's responsibility to ensure that method interferences caused by contaminants in solvents, reagents, glassware, and other sample processing hardware leading to discrete artifacts and/or elevated baselines in the chromatograms be minimized. In the extreme case of chronic contamination, blanks may have to be analyzed from each stage of the sample processing to determine the contamination source so it can be eliminated. In all cases where blank contamination exceeds the control limit a narrative comment must be made which documents the corrective actions taken.

4.14 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 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.

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 required that a matrix spike analysis be performed with each extraction batch. The minimum frequency for MS analysis is 1 each per 20 samples per matrix. This matrix spike sample is used to evaluate the matrix effect of the sample upon recovery of the analytes. The recovery of spike analytes is calculated as follows:

$$\%\text{Recovery} = \frac{MS - SA}{S} x100$$

where:
MS = concentration in spiked sample
SA = native concentration in unspiked sample
S = spike amount

4.15.1.2 The recovery criteria are detailed in 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.

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 to measure method precision. This

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

$$\%RPD = \frac{|\chi - \delta|}{(\chi + \delta)/2} x100$$

where:

χ = measured concentration for MS sample
δ = measured concentration for MSD sample

4.16.1.2 RPD control limits are detailed in the QC database, QC_DB.

4.16.2 Corrective action

4.16.3 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 Surrogate Recovery

4.17.1 Criteria

4.17.1.1 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

4.17.1.2 Detailed surrogate recovery control limits are tabulated in the QC database, QC_DB.

4.17.2 Corrective Action

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

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

5. Operation procedures

5.1 Chromatographic Conditions

5.1.1 The following general operating parameters are used on gas chromatographs to perform this method utilizing megabore (0.53 or 0.45mm) columns:

Carrier gas:	helium
Column flow:	8 mL/min
Make-up gas:	argon/methane (95%/5% High Purity grade)
Make-up flow:	70 mL/min
Injector:	Grob-type, splitless
Injector temperature:	205°C
Injection:	splitless
Injection volume:	2µL (split - 1 µL per column)
Initial temperature:	150°C
Initial hold time:	0.5 min
Temperature ramp:	4°C per min
Final temperature:	275°C
Final hold time:	9 min
EC detector temperature:	350°C

These GC conditions should be optimized for analyte separation and sensitivity with a particular pair of columns and detectors. Once optimized, the same conditions must be used for the analysis of all standards, samples, blanks and spikes.

The following general operating parameters are used on gas chromatographs to perform this method utilizing capillary (0.25mm) columns:

Carrier gas:	helium
Linear velocity:	28 cm/sec
Make-up gas:	argon/methane (95%/5% High Purity grade)
Make-up flow:	70 mL/min
Injector:	Grob-type, splitless
Injector temperature:	250°C
Injection:	splitless
Injection volume:	1µL (split - 0.5 µL per column)
Initial temperature:	110°C
Initial hold time:	0.5 min
Initial Temperature ramp:	25°C per min
Final temperature:	150°C
Hold time:	0. min
Temperature ramp A:	12°C per min
Final temperature:	260°C
Hold time:	0. min
Temperature ramp B:	15°C per min
Final temperature:	320°C
Hold time:	3.5 min
EC detector temperature:	350°C

These GC conditions should be optimized for analyte separation and sensitivity with a particular pair of columns and detectors. Once optimized, the same conditions must be used for the analysis of all standards, samples, blanks and spikes.

5.2 Sample Analysis

5.2.1 Analysis sequence

5.2.1.1 See Appendix II for a detailed analysis injection sequence.

5.2.2 Compound Identification

5.2.2.1 Compounds are tentatively identified if a peak elutes in the retention time window characteristic of that compound on the column 1. To confirm the presence of that compound in the sample extract, the peak must also elute in its characteristic retention time window on a second column. Retention time windows are established as previously described and the absolute retention times are updated each QC period. Compounds can only be identified if the ICV and CCV criteria previously detailed are strictly adhered to.

5.2.2.2 The experienced analyst's judgment weighs heavily in evaluating chromatograms for compound identification. For instance, the retention times of surrogate compounds may be outside their expected windows due to sample matrix effects. The analyst may decide to re-adjust the target analyte's retention time windows on an ad hoc basis based on such an observed shift. This can occur only on a sample-specific basis and is used when the analyst examining the data suspects that a retention time shift has occurred. If this is done, it must be fully documented in the case narrative notes.

5.2.2.3 Identification of multicomponent analytes occurs when the retention times and ratios of each of the multicomponent peaks present in the sample match the peaks chosen for the particular multicomponent analyte in the initial calibration. These same conditions must be met on both columns in order to confirm the presence of the multicomponent analyte. The experienced analyst's judgment weighs heavily in evaluating the patterns of multicomponent analytes with regards to weathering and matrix interferences.

5.2.3 Compound Quantitation

Target compound concentrations are calculated using the following equations:

5.2.3.1 Aqueous samples

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

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

where:

A_x = Response for the analyte in the extract, in area or height units.
CF_m = Multi-point average CF
V_i = Volume of extract injected, µL.
D = Dilution factor of extract. The final result of an algebraic multiplication of the ratio of all dilution final volumes to initial volumes. For example, if and extract was

diluted 100 µL to 1000 µL and subsequently diluted an additional 100 µL to 1000 µL, the expression would be: (1000/10) * (1000/10) = 100 * 100 = 10,000. If no dilution was made, D = 1.

V_t = Volume of total extract, µL.

V_s = Initial sample size, mL.

The reported concentration for multicomponent analytes is calculated is based on an average of the concentrations determined for each of the peaks chosen for calibration.

5.2.3.1.2 To report concentrations in alternate units, apply an appropriate factor:

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

5.2.3.2 Non-aqueous samples

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

$$Conc.(\mu g / kg) = \frac{(A_x)(V_t)(D)}{(CF_m)(V_i)(W)(TS/100)}$$

where:

W = Weight of sample extracted or purged, grams.

TS = Total solids, percent.

The reported concentration for multicomponent analytes is based on an average of the concentrations determined for each of the peaks chosen for calibration.

5.2.4 Sample Dilution

If the responses in the sample chromatogram exceed the calibration range of the system, dilute the extract and reanalyze. The dilution should be made so that the concentration of the analyte requiring dilution is in the mid to upper calibration range.

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

6.3 Sample Result Reports

6.3.1 Data Qualifying Flags

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

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

6.4 Control Chart(s)

6.4.1.1 The recovery values for gamma-BHC, Heptachlor, Aldrin, Aroclor 1260, TCMX, and DCB in the Blank Spike are plotted on control charts.

7. References

1). U.S. EPA Contract Laboratory Program Contract, Exhibit D, Section III, March 1990

2). U.S. EPA SW846 Test Methods for Evaluating Solid Waste, Method 8000B, Gas Chromatography, Revision 2, December 1996.

3). U.S. EPA SW846 Test Methods for Evaluating Solid Waste, Method , Organochlorine Pesticides and PCBs, Method 8081, Revision 1, December 1996 .

APPENDIX I

Standard Solution Concentrations, µg/L

Compound	STD1	STD2	STD3	STD4	STD5
alpha-BHC	5.0	10.0	20.0	40.0	80.0
beta-BHC	5.0	10.0	20.0	40.0	80.0
delta-BHC	5.0	10.0	20.0	40.0	80.0
gamma-BHC (Lindane)	5.0	10.0	20.0	40.0	80.0
alpha-Chlordane	5.0	10.0	20.0	40.0	80.0
gamma-Chlordane	5.0	10.0	20.0	40.0	80.0
Heptachlor	5.0	10.0	20.0	40.0	80.0
Aldrin	5.0	10.0	20.0	40.0	80.0
Heptachlor epoxide	5.0	10.0	20.0	40.0	80.0
Endosulfan I	5.0	10.0	20.0	40.0	80.0
Dieldrin	10.0	20.0	40.0	80.0	160
4,4'-DDE	10.0	20.0	40.0	80.0	160
Endrin	10.0	20.0	40.0	80.0	160
Endosulfan II	10.0	20.0	40.0	80.0	160
4,4'-DDD	10.0	20.0	40.0	80.0	160
Endosulfan sulfate	10.0	20.0	40.0	80.0	160
4,4'-DDT	10.0	20.0	40.0	80.0	160
Methoxychlor	50.0	100	200	400	800
Endrin aldehyde	10.0	20.0	40.0	80.0	160
Endrin ketone	10.0	20.0	40.0	80.0	160
Isodrin	12.5	25.0	50.0	100	200
Chlordane - technical					1000
Toxaphene			500		
Aroclor-1016	100	250	500	1000	2000
Aroclor-1221			500		
Aroclor-1232			500		
Aroclor-1242			500		
Aroclor-1248			500		
Aroclor-1254			500		
Aroclor-1260	100	250	500	1000	2000
2,4,5,6-tetrachloro-m-xylene	5.0	10.0	20.0	40.0	80.0
Decachlorobiphenyl	10.0	20.0	40.0	80.0	160

Appendix I (Continued)

Calibration Stock Solutions, mg/L

Compound	Mix	Conc
alpha-BHC	A	5.0
beta-BHC	B	5.0
delta-BHC	B	5.0
gamma-BHC (Lindane)	A	5.0
alpha-Chlordane	B	5.0
gamma-Chlordane	B	5.0
Heptachlor	A	5.0
Aldrin	B	5.0
Heptachlor epoxide	B	5.0
Endosulfan I	A	5.0
Dieldrin	A	10.0
4,4'-DDE	B	10.0
Endrin	A	10.0
Endosulfan II	B	10.0
4,4'-DDD	A	10.0
Endosulfan sulfate	B	10.0
4,4'-DDT	A	10.0
Methoxychlor	A	50.0
Endrin aldehyde	B	10.0
Endrin ketone	B	10.0
Isodrin	A	25
Chlordane - technical	TChlor	1000
Toxaphene	Tox	500
Aroclor-1016	AR1660	100
Aroclor-1221	AR1221	100
Aroclor-1232	AR1232	100
Aroclor-1242	AR1242	100
Aroclor-1248	AR1248	100
Aroclor-1254	AR1254	100
Aroclor-1260	AR1660	100

*-The single component pesticides are contained in 2 separate mixes (A and B) when utilizing megabore (0.53 or 0.45mm ID) columns. These same components are combined into 1 mix when utilizing capillary (0.25mm ID) columns.

Breakdown Check Solution (CLP PEM solution), µg/L

Compound	Conc
4,4'-DDT	100
Endrin	50

Surrogate Stock Solution, mg/L

Compound	Conc.
2,4,5,6-tetrachloro-m-xylene	10.0
Decachlorobiphenyl	10.0

APPENDIX II

Analysis Sequence

Injection	Sample
	hexane rinse
1	breakdown check standard (PEM)
2	ICV standard INDAM
3	ICV standard INDBM
4	ICV standard multicomponents(for PCB only analysis - can replace above 2 stds)
5	up to 10 subsequent sample or QC extracts
15	solvent rinse or IBLK (optional - not required)
16	CCV standard INDAM
17	CCV standard INDBM
18	CCV standard multicomponents (for PCB only analysis - can replace above 2 stds)
19	up to 10 subsequent sample or QC extracts
last	CCV standard INDAM
last	CCV standard INDBM
last	CCV standard multicomponents (for PCB only analysis - can replace above 2 stds)

APPENDIX III

Data Packet Order

I. QC SUMMARY

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

II. SAMPLE DATA:

_____ Organic Analysis Data Sheet
_____ Sample Confirmation Worksheet
_____ Chromatograms, column 1
_____ Chromatograms, column 2
_____ Chromatographic Report, column 1
_____ Chromatographic Report, column 2

III. STANDARD DATA:

_____ Linearity Report

Linearity Standards:
_____ Chromatograms, column 1
_____ Chromatograms, column 2
_____ Chromatographic Report, column 1
_____ Chromatographic Report, column 2

Continuing Calibration Standards:
_____ CCV Report
_____ Chromatograms, column 1
_____ Chromatograms, column 2
_____ Chromatographic Report, column 1
_____ Chromatographic Report, column 2

APPENDIX III, continued

Other Standards Used to Support Sample Data and Instrument Blanks

V. Raw QC Data:

_____ Method Blank
_____ Chromatograms, column 1
_____ Chromatograms, column 2
_____ Chromatographic Report, column 1
_____ Chromatographic Report, column 2

_____ Blank Spike
_____ Chromatograms, column 1
_____ Chromatograms, column 2
_____ Chromatographic Report, column 1
_____ Chromatographic Report, column 2

_____ Matrix Spike
_____ Chromatograms, column 1
_____ Chromatograms, column 2
_____ Chromatographic Report, column 1
_____ Chromatographic Report, column 2

_____ Matrix Spike Duplicate
_____ Chromatograms, column 1
_____ Chromatograms, column 2
_____ Chromatographic Report, column 1
_____ Chromatographic Report, column 2

APPENDIX III, continued

V. Bench Sheets

_____ Injection Sequence
_____ Target Method
_____ Extraction Bench Sheets
_____ Miscellaneous Work Sheets. i.e. %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 8081 QA Requirements and Corrective Actions:

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Initial Calibration	5 calibration stds, for all single and multi-components, %RSD ≤20% for each analyte or averaged across all analytes.	5 calibration stds, for all single and multi-components, %RSD ≤20% for each analyte or averaged across all analytes.	After performing major instrument maintenance or when persistent difficulties meeting CCV criteria occur.	recalibrate or narrate faults if possible	narrative
Initial Calibration Verification	Must be ±15% D for each analyte or averaged across all analytes.	Must be ±15% D for each analyte or averaged across all analytes.	at start of each new analytical sequence, or daily if RTs shift	rerun with new ICV, instrument maintenance, &/or recalibrate (or narrate if possible)	narrative
Continuing Calibration Verification	Must be ±15% D for each analyte or averaged across all analytes.	Must be ±15% D for each analyte or averaged across all analytes.	Every 10 samples	rerun with new ICV, instrument maintenance, &/or recalibrate (or narrate if possible)	narrative
Breakdown	Endrin and DDT breakdown must be <=15%	Endrin and DDT breakdown must be <=15%	At the beginning of each sequence.	Rerun, instrument maintenance	narrative
Method Blank	Must be below MDL	Must be below reporting limit	every analytical batch or 1 per 20 samples	Report quantitated contaminants with a "B" flag, or re-extract if necessary	case narrative and corrective action form if re-extracted
Surrogate Recovery	Limits to be determined by the lab	listed in QC database	every sample	if other QC is in control, narrate; otherwise re-extract	case narrative and corrective action form if re-extracted
Matrix Spike Recovery	Limits not specified	listed in QC database	every analytical batch or 1 per 20 samples	if other QC is in control, narrate; otherwise re-extract	case narrative and corrective action form if re-extracted
MS/MSD RPD	Limits not specified	listed in QC database	every analytical batch or 1 per 20 samples	if other QC is in control, narrate; otherwise re-extract	case narrative and corrective action form if re-extracted
Blank Spike Recovery	Limits not specified	listed in QC database	every analytical batch or 1 per 20 samples	if other QC is in control, narrate; otherwise re-extract	case narrative and corrective action form if re-extracted
Standard Reference Material (SRM) Recovery	none required	none required	only if requested	reanalyze or re-extract and reanalyze SRM	case narrative and corrective action form if re-extracted

APPENDIX V

Compound Elution order on DB5 and DB608 Megabore (0.45 or 0.53 mm ID) Columns

DB5	DB608
Tetrachloro-m-xylene	Tetrachloro-m-xylene
alpha-BHC	alpha-BHC
beta-BHC	gamma-BHC (Lindane)
gamma-BHC (Lindane)	beta-BHC
delta-BHC	Heptachlor
Heptachlor	delta-BHC
Aldrin	Aldrin
Isodrin	Isodrin
Heptachlor epoxide	Heptachlor epoxide
gamma-Chlordane	gamma-Chlordane
Endosulfan I	alpha-Chlordane
alpha-Chlordane	Endosulfan I
4,4'-DDE	4,4'-DDE
Dieldrin	Dieldrin
Endrin	Endrin
Endosulfan II	4,4'-DDD
4,4'-DDD	Endosulfan II
Endrin aldehyde	4,4'-DDT
Endosulfan sulfate	Endrin aldehyde
4,4'-DDT	Endosulfan sulfate
Endrin ketone	Methoxychlor
Methoxychlor	Endrin ketone
Decachlorobiphenyl	Decachlorobiphenyl

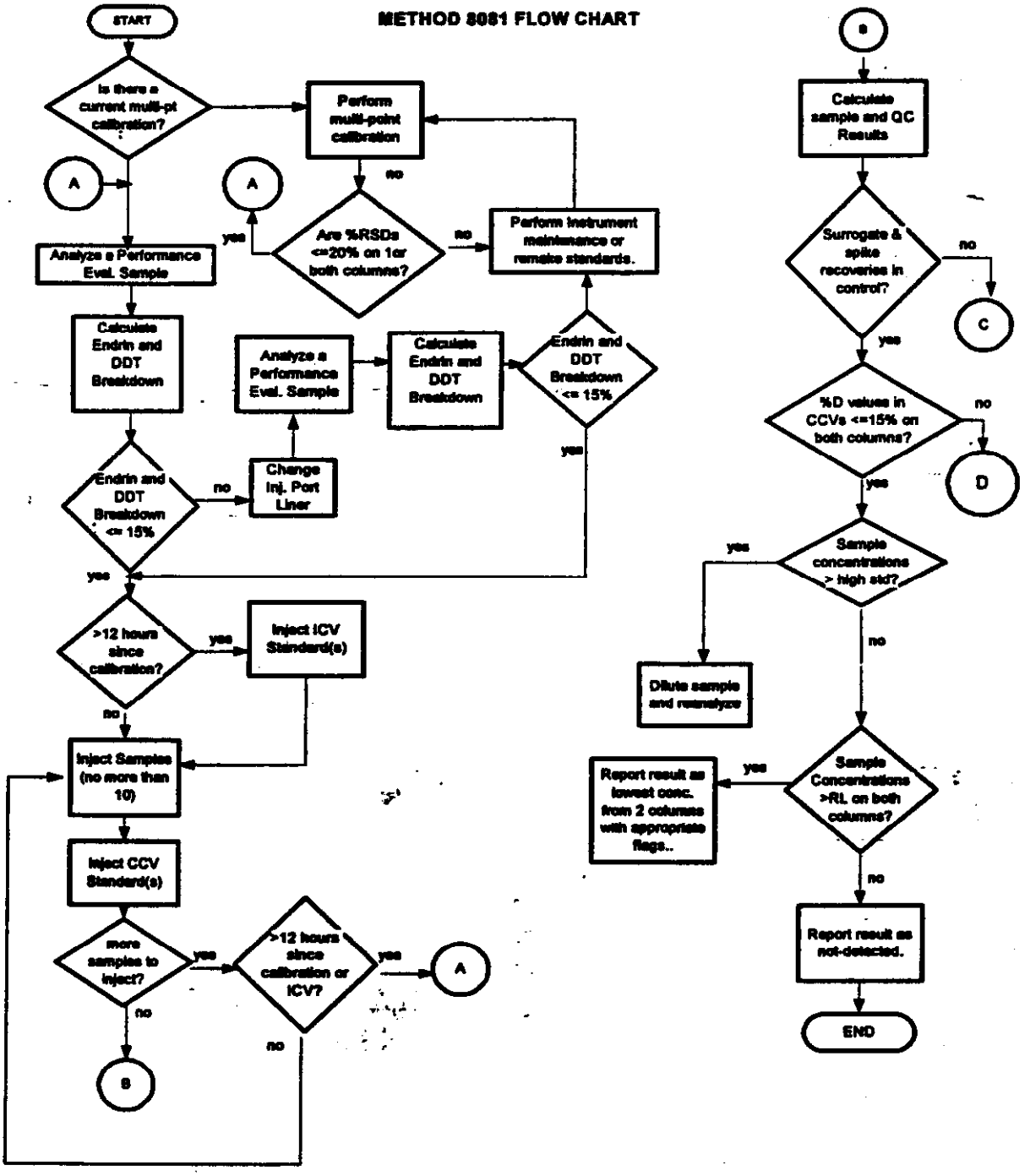
APPENDIX VI

Compound Elution order on DBXLB and DB17ms Capillary (0.25 mm ID) Columns

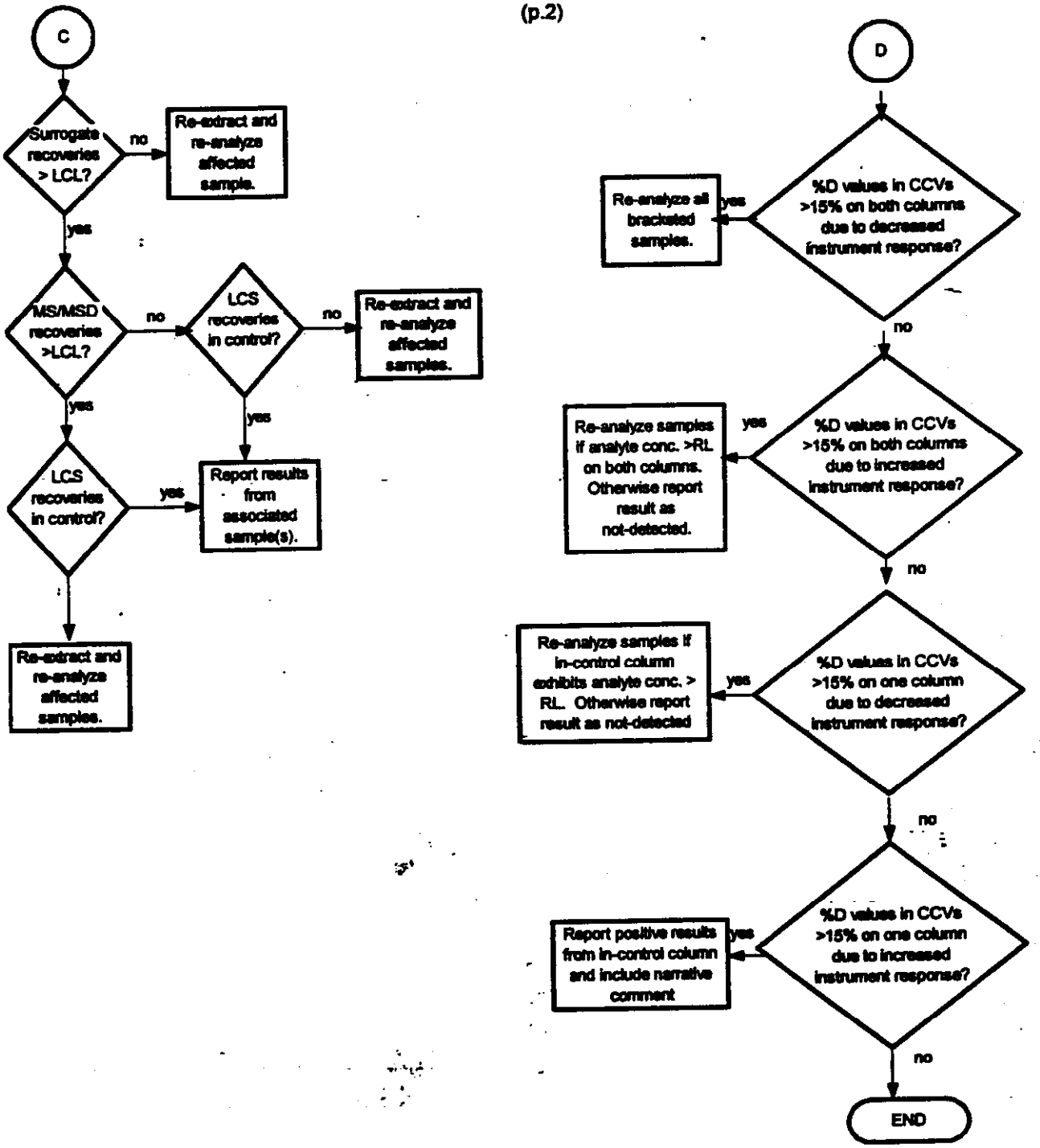
DBXLB	DB17ms
Tetrachloro-m-xylene	Tetrachloro-m-xylene
alpha-BHC	alpha-BHC
gamma-BHC (Lindane)	gamma-BHC (Lindane)
beta-BHC	beta-BHC
delta-BHC	Heptachlor
Heptachlor	delta-BHC
Aldrin	Aldrin
Isodrin	Isodrin
Heptachlor epoxide	Heptachlor epoxide
gamma-Chlordane	gamma-Chlordane
alpha-Chlordane	alpha-Chlordane
Endosulfan I	Endosulfan I
4,4'-DDE	4,4'-DDE
Dieldrin	Dieldrin
Endrin	Endrin
4,4'-DDD	4,4'-DDD
Endosulfan II	Endosulfan II
Endrin aldehyde	4,4'-DDT
4,4'-DDT	Endrin aldehyde
Endosulfan sulfate	Endosulfan sulfate
Methoxychlor	Methoxychlor
Endrin ketone	Endrin ketone
Decachlorobiphenyl	Decachlorobiphenyl

APPENDIX VII

Flow Chart



METHOD 8081 FLOW CHART
(p.2)



LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-8276

Determination of Semivolatile Organic Compounds by SW-846 Method 8270C

Revision history:

Number	Date
0	02/05/98
1	02/17/99

Written by: Galina Gringer Date: 2/17/99
Galina Gringer, GC/MS Semivolatile Chemist

Approved by: Harry Romberg Date: 2-17-99
Harry Romberg, QA Officer

Approved by: Kathy Kreps Date: 2-23-99
Kathy Kreps, Laboratory Director

Controlled Document

No. 20 Assigned to: TETRA

TABLE OF CONTENTS

1. INTRODUCTION AND SCOPE 4

1.1 METHOD DESCRIPTION 4
1.2 METHOD DEVIATIONS & COMMENTS 4
1.3 SAMPLE COLLECTION, SAMPLE STORAGE, HOLDING TIMES 5
1.4 DEFINITION OF TERMS 5

2. EQUIPMENT LIST AND STANDARDS 8

2.1 CHROMATOGRAPHIC SYSTEM 8
2.2 STANDARDS 9

3. SAFETY PRECAUTIONS 13

3.1 ROUTINE SAFETY PRECAUTIONS 13
3.2 WASTE DISPOSAL 14

4. OPERATION PROCEDURES 14

4.1 ANALYTICAL CONDITIONS 14
4.2 METHOD DETECTION LIMIT STUDY 15
4.3 METHOD REPORTING LIMITS 15
4.4 METHOD VALIDATION 15
4.5 DAILY INSTRUMENT MAINTENANCE 15
4.6 INSTRUMENT TUNING 16
4.7 INITIAL MULTI-POINT CALIBRATION 16
4.8 CONTINUING CALIBRATION VERIFICATION 16
4.9 SAMPLE ANALYSIS 17

5. REPORTS 21

5.1 DATA PACKET ORGANIZATION 21
5.2 QUALITY CONTROL REPORTS 21
5.3 SAMPLE RESULT REPORTS 21

6. QUALITY CONTROL 22

6.1 GENERAL ISSUES 22
6.2 INITIAL CALIBRATION 22
6.3 CONTINUING CALIBRATION VERIFICATION 24
6.4 METHOD BLANK 24
6.5 QC CHECK SAMPLE (LCS) 25
6.6 MATRIX SPIKE 26
6.7 MATRIX SPIKE DUPLICATE 27
6.8 SURROGATE RECOVERY 28

7. REFERENCES 29

APPENDIX I 30

CALIBRATION STANDARD SOLUTION CONCENTRATIONS, NG/µL 30

APPENDIX II 33
DFTPP TUNING CRITERIA 33
APPENDIX III 34
DATA PACKET ORDER LIST 34
APPENDIX IV 35
INITIAL CALIBRATION CRITERIA 35
APPENDIX V 36
CONTINUING CALIBRATION VERIFICATION CRITERIA 36
APPENDIX VI 37
METHOD BLANK CONTROL LIMITS 37
APPENDIX VII 38
METHOD 8270 FLOW CHART 38
APPENDIX VIII 39
METHOD 8270C QA REQUIREMENTS AND CORRECTIVE ACTIONS 39
APPENDIX IX 41
APPENDIX IX CALIBRATION STANDARD SOLUTION CONCENTRATIONS, NG/µL 41
APPENDIX X 43
ANALYTES AMENABLE TO ANALYSIS BY METHOD 8270C 43
APPENDIX XI 47
SEMIVOLATILE INTERNAL STANDARDS WITH CORRESPONDING ANALYTES ASSIGNED FOR QUANTITATION 47
APPENDIX XII 49
COMMENTS ON ANALYSIS OF APPENDIX IX SEMIVOLATILE COMPOUNDS 49

1. Introduction and Scope

1.1 Method Description

1.1.1 This method is used for the determination of semivolatile organic analytes in aqueous, soil, sediment, and other matrices. This SOP follows SW-846 Method 8270C except for the specific deviations listed below or outlined in a project's specific QAPP.

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

1.2 Method Deviations & Comments

1.2.1 The following items represent deviations and comments of Method 8270C, as published, which are followed as standard operating procedure in the performance of this method at Laucks:

1.2.1.1 Method 3640A (Gel Permeation Chromatography cleanup) is utilized routinely for all soil/sediment sample extracts. GPC cleanup is performed on aqueous sample extracts as deemed necessary.

1.2.1.2 The initial calibration has been increased from five to six concentration levels in order to demonstrate linearity for all target analytes and to provide a low-level standard that will act as the reporting limit as outlined in the method.

1.2.1.3 Surrogate compounds 2-chlorophenol-d4 and 1,2-dichlorobenzene-d4 are used routinely in addition to the six surrogate compounds specified in Method 8270C. These additional surrogates are advisory only.

1.2.1.4 The concentration of internal standards added to all sample extracts and calibration standards has been decreased from 40 ng/µL per compound to 20 ng/µL in order to accommodate 2 µL injection volumes as allowed by the method. This adjustment keeps the total nanograms injected the same and prevents overloading the analytical column.

1.2.1.5 During daily maintenance procedure it is necessary to break off the first 10 cm (or more) of the capillary column, which results in shifting of the retention time of all target analytes and internal standards as well. When daily calibration verification standard is run, all retention times have been updated. Therefore, there is no reason to recalibrate the system if RTs for any internal standards in the CCV changed more than 30 seconds

from that in the mid-point calibration standard level of the most recent initial calibration sequence, as stated in the Method 8270C

1.2.1.6 Although it's not 8270C Method requirement, internal standard retention times and responses in all samples, spikes, and blanks have been monitored. Acceptable retention times used for internal standards in all analyses is ± 0.50 minutes relative to the daily CCV standard. The acceptable area counts for IS are -50% to +100% of the IS in the CCV.

1.2.1.7 Lauck's uses a relative response factor for all analytes. The method specifies that if the average RSD of all analytes for the initial calibration is ≤15%, then the RRF may be used for individual analytes with RSDs >15%. This method option will be used if any analyte's RSD exceeds 15% in the initial calibration.

1.2.1.8 CLP Statement of Work, OLM03.1, DFTPP tuning criteria from has been substituted as allowed by the method. Tuning criteria is listed in Appendix II.

1.2.1.9 All standards are stored at less than -10°C or by the manufacturer's recommendation. Sample extracts are stored at 4°C ±2°C.

1.2.1.10 The surrogate and matrix spikes will be added to the sample such that the final amount injected from normally concentrated samples is 100 ng for the base/neutral compounds and 150 ng for the acids. Acids will not be injected at 200 ng per injection to prevent exceeding the highest calibration standard of 160 ng.

1.3 Sample Collection, Sample Storage, Holding Times

1.3.1 Samples are normally collected in glass containers with Teflon-lined caps. All samples and sample extracts are stored at 4°C ± 2°C. Water samples must be extracted within 7 days of collection. Soil samples must be extracted within 14 days of collection. All sample extracts must be analyzed within 40 days of sample preparation.

1.4 Definition of Terms

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

Method Blank Spike	A background free matrix (DIW for water, clean sand for soils/sediments) to which known amounts or target analytes and surrogates are added each time sample extracts are prepared. Blank spikes are required on all HAZWRAP and NFESC work. In the context of this SOP, a blank spike is the same as a QC check standard.
CCC	Calibration Check Compound. A compound of analytical interest whose RRF in the CCV is compared to the average RRF from the initial calibration. The % difference must be less than the value specified in the method for the CCV to be considered valid. CCCs must also meet maximum %RSD criteria in an initial calibration.
CCV	Continuing calibration verification. This is the same acronym used in the CLP program. This is a standard injected at some prescribed frequency during the analysis sequence to determine whether the instrument has remained in calibration.
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.
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.
IPCS	Instrument Performance Check Solution. A solution containing at a minimum DFTPP, pentachlorophenol, benzidine, and p,p'-DDT. The IPCS is analyzed at the start of a 12 hour QC period in order to verify DFTPP tuning criteria and evaluate column tailing factors. There are no specified tailing factor criteria in the published method, however, tailing factors should not exceed 4.0 .

Internal Standard	A compound added to every standard, blank, matrix spike, matrix spike duplicate, and sample extract at a know concentration, prior to analysis. Internal standards are used as the basis for quantitation of target compounds.
MDL	Method detection limit. The lowest concentration in a sample which will yield a positive result that is greater than zero at a known level of confidence. MDLs are empirically determined by Laucks.
MDL Standard	Method detection limit standard. A standard prepared so that the concentrations of the target analytes are approximately 4x the empirically determined MDLs on an extract basis. This standard is used to verify that the instrument is capable of detecting the target analytes on an ongoing basis.
QC Period	Quality control period. An analysis sequence initiated by the injection of one or more standards, followed by sample extracts. A QC period is 12 hours starting with the injection of the CCV standard or DFTPP performance evaluation.
RF	Response Factor. The measure of the mass spectral response of an analyte compared to its internal standard. Response factors are determined by analysis of standards and are used in the calculation of concentrations of analytes in samples. RF is determined by the following equation:

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

where:

A_x - Area of target analyte primary ion
C_{is} - Concentration of internal standard
A_{is} - Area of internal standard primary ion
C_x - Concentration of target analyte

RSD or %RSD	Relative standard deviation or percent relative standard deviation. The ratio of the standard deviation of a set of values to the mean of the set of values. A measure of the similarity of the values one to another.
RT	Retention time. The time (in minutes) at which a target analyte elutes from the GC column.
Sequence	A set of sample extracts and standard solutions injected into an instrument in a chronologically continuous group. See also QC period.
SPCC	System Performance Check Compound. A compound of analytical interest whose relative response factor (RRF) is monitored in the CCV for signs of chromatographic degradation. SPCCs generally have low RRFs and tend to decrease in response as the chromatographic system begins to deteriorate. They must meet a minimum RRF for initial and continuing calibrations.
EICP	Extracted ion current profile-a plot of ion abundance versus time (or scan number) for ion(s) of specified mass(es).
Surrogate	Compounds added to every standard, blank, matrix spike, matrix spike duplicate, and sample extract at a known concentration; used to evaluate extraction and analytical efficiency by measuring recovery. Surrogates are brominated, fluorinated, or isotopically labeled compounds not expected to be detected in environmental samples.

2. Equipment List and Standards

2.1. Chromatographic System

Gas Chromatograph:	Hewlett Packard 5890 I or II
Carrier Gas:	Helium 99.999% (ultra purity) or better
Column:	30 m x 0.25 mm x 0.25 μ capillary column (Restek RTX-5 or equivalent)
Automatic Sampler:	Hewlett Packard 7672A with 19405A and 3396A controllers
GC/MS Interface:	Capillary direct to the ion source of the mass spectrometer, fixed temperature

Mass Spectrometer:	Hewlett Packard 5970B
Data System:	Teknivent
Miscellaneous:	Assorted syringes, vials, caps, septa, injection port liners, ferrules, etc.
Note:	All of the above equipment may be substituted with equivalent or better equipment

2.2 Standards

2.2.1 Preparation of Semivolatile Standards

2.2.1.1 All standards prepared from this operating procedure must be logged into one of two standards preparation logbooks. One is maintained for stock solutions prepared from neat chemicals; the other is maintained for all working solutions. These logbooks are kept in the GC/MS semivolatile working area. When a standard is made, a solution number is assigned to it. This solution number is unique and will be used to track and identify the standard every time it is analyzed.

2.2.1.2 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 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.

2.2.1.3 All standards must also be verified both qualitatively and quantitatively in order to satisfy EPA requirements for traceability. This may be accomplished by purchasing solutions which have been fully documented by a commercial vendor.

2.2.2 Preparation of Internal Standard Solution (ABN IS MIX @ 2000 ug/mL).

Internal standards
1,4-Dichlorobenzene-d4
Naphthalene-d8
Acenaphthene-d10
Phenanthrene-d10
Chrysene-d12
Perylene-d12

2.2.2.1 Commercially prepared and certified internal standard solution is purchased at a concentration of 2000 µg/mL and is used undiluted. This standard is monitored for degradation by evaporative losses. The standard should be replaced when the area counts increase more than 15% from when it was freshly opened. This standard is kept at -10°C until put into use. Once opened, the standard is kept at room temperature to avoid having the less soluble compounds fall out of solution. A new ampule of internal standard is used with a new CCV standard, which is prepared on a weekly basis.

2.2.3 Preparation of Surrogate Standards

Surrogate compounds
2-Fluorophenol
Phenol-d5
2-Chlorophenol-d4
2,4,6-Tribromophenol
1,2-Dichlorobenzene-d4
Nitrobenzene-d5
2-Fluorobiphenyl
p-Terphenyl-d14

2.2.3.1 Surrogate stock solutions are prepared by dissolving 100 mg of each analyte in 10 mL of methanol resulting in a stock solution with a concentration of 10,000 µg/mL. The one exception is p-terphenyl-d14 which is prepared by dissolving 50 mg in 10 mL of 50:50 benzene/methylene chloride for a final concentration of 5000 µg/mL.

2.2.3.2 An aliquot of each stock solution is diluted 1:200 (1:100 for p-terphenyl-d14) in methylene chloride and analyzed by the GC/MS department before they are incorporated into working solutions. The stocks must be within 80% - 120% of the expected value of 50 ng/µL to be considered usable.

2.2.3.3 5 mL of each of the base/neutral surrogate stock solutions (10 mL of the p-terphenyl-d14) and 7.5 mL of each of the acid surrogate solutions are mixed and diluted in methanol to a final volume of 500 mL to make a working solution of 100 µg/mL for the base/neutral surrogates and 150 µg/mL for the acid surrogates. An aliquot of the working solution is diluted 1:5 in methylene chloride and analyzed by the GC/MS department. The working solution must be within 80% - 120% of the expected values of 30 ng/µL for the acid surrogates and 20 ng/µL for the base/neutral surrogates before they are put into use by the extractions department.

2.2.3.4 Commercially prepared and certified surrogate solution may be purchased and used in place of the above described solutions at the discretion of the laboratory.

2.2.4 Preparation of Matrix Spike Standards

Matrix spike compounds
Phenol
2-Chlorophenol
1,4-Dichlorobenzene
N-Nitroso-di-n-propylamine
1,2,4-Trichlorobenzene
4-Chloro-3-methylphenol
Acenaphthene
2,4-Dinitrotoluene
4-Nitrophenol
Pentachlorophenol
Pyrene

2.2.4.1 Stock and working solutions of all matrix spike compounds utilized are prepared and verified in the same manner as surrogate standards. The final concentration of the acid compounds will be 150 µg/mL and the base/neutral compounds will be at a concentration of 100 µg/mL.

2.2.4.2 Commercially prepared and certified spiking solution may be purchased and used in place of the above described solution.

2.2.5 Preparation of DFTPP Solution

2.2.5.1 Prepare a solution of DFTPP (Decafluorotriphenylphosphine) at a concentration of 5000 µg/mL in acetone. Store this solution in amber screw-cap vials in the freezer. The vial in use may be stored at room temperature.

2.2.5.2 Commercially prepared and certified DFTPP solution may be purchased and used in place of the above described solution. This solution and working standards made from it are kept at -10°C.

2.2.6 Preparation of Calibration Standards

2.2.6.1 Calibration standards are prepared in methylene chloride from stock solutions which are purchased from a commercial source (e.g., Supelco or Restek). If an analyte required for calibration is not present in an available mixed solution, laboratory-prepared stock solutions which have been verified by GC/MS may be used. Laboratory prepared stock solutions should be tested against independent reference standards when they are available.

2.2.6.2 Calibration standards are prepared at six concentration levels (5, 10, 25, 40, 60 and 80 ng/µL). Each calibration standard contains all compounds of interest, surrogates, and internal standards. The internal standards are added so that they are present in all calibration standards at a concentration of 20 ng/µL each.

2.2.6.3 Through repetitive preparation of mixed semivolatile standards, it has been documented that benzidine is unstable when combined in working solutions with other semivolatile target compounds. Therefore, benzidine standards are prepared individually in the same manner as the multi-component stock and standard solutions.

2.2.7 Preparation of ABN200 stock solution

2.2.7.1 Assemble the following solutions in order to prepare a combined stock solution which will contain all analytes of interest at a concentration of 200 µg/mL each:

Analyte Mix	**Vendor**	**Stock #**	**Mix #** [(a)]
TCL Base-Neutrals Mix1	Supelco	#4-8900	(1)
TCL Base-Neutrals Mix2	Supelco	#4-8120	(2)
TCL Benzidines Mix	Supelco	#4-8906	(3)
TCL Hazardous Substances Mix1	Supelco	#4-8907	(4)
TCL Hazardous Substances Mix2	Supelco	#4-8908	(5)
TCL Phenols Mix	Supelco	#4-8904	(6)
TCL Polynuclear Aromatic Hydrocarbons Mix	Supelco	#4-8905	(7)
Acid Surrogate Mix	Supelco	#4-7261	(8)
B/N Surrogate Mix	Supelco	#4-7262	(9)
Pyridine	Supelco	#48305-U	(10)

(a) Refers to Appendix I standard solution information

Note: 1,2-diphenylhydrazine is detected as azobenzene. Azobenzene may be substituted for 1,2-diphenylhydrazine as it is in many commercially purchased calibration mixes.

Note: If performing additional analytes such as Appendix IX, the calibration stock and solutions are prepared separately. See Appendix IX of this SOP for details.

2.2.7.2 The vendor and catalog numbers provided are for reference only. Other vendor's certified solutions may be substituted.

2.2.7.3 Combine appropriate amounts of all solutions in a clean, silanized volumetric flask (10 mL capacity) so that all analytes are present at 200 µg/mL. Dilute to volume with methylene chloride, stopper the flask and mix well. Record all information in the working standards logbook and transfer the contents of the volumetric flask into silanized amber screw-cap vials. Store this solution in the freezer when not in use. The

vial must be marked with the logbook name, standard type, preparation date, solvent used, and expiration date.

2.2.8 Preparation of working calibration standards

2.2.8.1 To prepare working calibration standards, add the amounts listed below (in µL) of ABN200 stock solution, internal standard solution (ABN IS MIX @ 2000 ug/mL) and methylene chloride to clean vial inserts and use within one week.

	Amount of Standard ABN200 in µl	**Amount of Internal Standard in µl**	**Amount of CH_2Cl_2 in µl**
Working ABN 5	5	2	193
Working ABN 10	10	2	188
Working ABN 25	25	2	173
Working ABN 40	40	2	158
Working ABN 60	60	2	138
Working ABN 80	80	2	118

2.2.8.2 Log into the working solutions logbook all of the above information as the standards are made. Store all standards at less than -10°C for up to one week when not in use.

2.2.8.3 The working ABN 25 standard is made in larger quantity because it is used every day as a calibration check. It will be necessary to prepare a fresh ABN 25 on a weekly basis.

2.2.8.4 Calibration stock solutions which are received sealed in ampules from the manufacturer are useable up to their manufacturer's expiration date. The mixed ABN200 stock solution is stable for up to 6 months or 1 year when promptly ampuled. Working calibration standards may be used for 1 week.

3. Safety precautions

3.1 Routine Safety Precautions

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

3.1.2 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 Almost all of the analytes under consideration are known or suspected carcinogens. Analysts should wash their hands after using any standard, solvent or sample extract. Additionally, a respirator should be worn or a fume hood utilized for extremely hazardous compounds or very dirty extracts.

3.2 Waste disposal

3.2.1 All waste solvents, expired standards and old extracts should be disposed of in the solvent waste can located in the prep area fume hood. Consult the laboratory SOP for more detail on waste disposal.

4. Operation procedures

4.1 Analytical Conditions

4.1.1 Chromatographic Conditions

Initial Temperature:	30°C
Initial Time:	4 minutes
Column Temperature Program:	8°C/minute
Final Temperature:	300°C
Final Time:	8 minutes
Injector Temperature:	275°C
Transfer Line Temperature:	280°C
Injector Purge Time Off:	42 seconds (0.7 minutes)
Injection Volume:	2 µL
Column Linear Velocity:	30-40 cm/sec (nominal 35 cm/sec measured at 30°C)

4.1.2 Mass Spectrometer Conditions

Electron Energy:	70 volts
Mass Range:	35 to 500 amu
Scan Time:	Not to exceed 1 second per scan
Scan Start Time:	4.5 minutes

4.2 Method Detection Limit Study

4.2.1 MDL studies are performed on an annual basis. This procedure is fully described in Laucks SOP on Determination of MDL's.

4.3 Method Reporting Limits

4.3.1 The method reporting limit for this method shall be set as described by method 8000B by using the lowest calibration standard as the method report limit. Values detected below this level will be reported but will be "J" flagged as outlined in Section 5.2 of this SOP.

4.4 Method Validation

4.4.1 Prior to the analysis of any samples, it is necessary to validate the method. A method validation study is performed in a similar manner to an MDL study with the exception that a minimum of 4 replicates are required and the concentration levels are typically higher. This procedure is fully described in Laucks SOP on Determination of QC limits.

4.5 Daily Instrument Maintenance

4.5.1 Daily instrument maintenance is required for good chromatography and proper calibration. The following steps must be undertaken as needed before the analysis of any standards or samples.

- Cool GC oven to 30°C
- Check background
- Remove injector septum and liner
- Remove column from injector
- Install a clean quartz liner
- Reinstall O-ring or replace if worn
- Install a new ferrule on the column
- Clip off 8-10 cm of the column. Check for proper cut
- Reinstall column in injector and adjust height
- Install injector cap and a new septum
- Clean and re-install the autosampler syringe
- Check the background again
- If background is okay, ramp the GC oven twice from 30°C to 300°C at 15°C/minute and hold 8 minutes

4.6 Instrument Tuning

4.6.1 The HP 5970B mass spectrometer uses FC-43 (PFTBA) as a mass calibration compound. Each instrument will require different ion ratios to pass the required DFTPP performance criteria. See Appendix II for the DFTPP tuning criteria. The following ratios are therefore approximate. Use manual tuning to tune the mass spectrometer.

Ion	% of ion 69
69	100%
131	25-35%
219	25-35%
414	1-3%

4.6.2 Ion peak widths should be in the 0.5 to 0.6 amu range. Excessive peak width can lead to loss of minor isotope peaks. Insufficient peak width can result in the loss of sensitivity. Ions for water, nitrogen, and oxygen should be <5% of ion 69. A copy of the PFTBA spectrum and tabular listing should be kept with the instrument historical file.

4.7 Initial Multi-Point Calibration

4.7.1 An instrument performance check solution containing DFTPP should be injected first in order to verify DFTPP tuning criteria, degradation, and column tailing factors. The spectrum of DFTPP must pass tuning criteria before any other standards are injected. Retuning of the instrument may be necessary to achieve this. Additionally, the DFTPP spectrum should be well within the performance criteria - i.e., no ion abundance should be borderline and the ratios should be routinely reproducible to insure a stable calibration.

4.7.2 Analyze the calibration standards following an acceptable DFTPP injection. Analyze standard solutions using six different concentration levels. The lowest concentration should reflect the current report limit being used. The highest concentration should define the upper usable working range of the detector. The initial calibration must meet the criteria outlined later in this SOP.

4.8 Continuing Calibration Verification

4.8.1 At the beginning of a QC period, analyze an instrument performance check solution. The DFTPP tuning criteria in Appendix II must be met prior to analyzing a CCV standard. If DFTPP tuning criteria are met, analyze a CCV standard. The standard analysis must meet the %D and minimum RRF criteria detailed later in this SOP.

4.9 Sample Analysis

4.9.1 Analysis sequence

4.9.1.1 In general, the method blank accompanying the samples is injected prior to the analysis of the samples. This is not a requirement, however. Samples are then injected until the end of the 12 hour clock. After 12 hours have expired, instrument maintenance is performed and another IPCS and CCV standard are analyzed and evaluated before sample analysis continues.

4.9.2 Extract Preparation

4.9.2.1 Remove sample extracts from the refrigerator and allow them to come to room temperature.

4.9.2.2 Transfer 200 µL of extract to a vial insert.

4.9.2.3 Add 2 µL of IS solution (ABN IS MIX @ 2000 ug/mL).

4.9.2.4 Cap the vial and mix well.

4.9.2.5 Place the vial onto the autosampler for analysis.

4.9.2.6 For dilutions, decide what dilution is necessary from previous data or analyst judgment. Make dilution and then add 2 µL of IS solution prior to capping vial. Mix well prior to analysis.

4.9.3 Compound Identification

4.9.3.1 Compounds are tentatively identified if a peak elutes within 0.5 minute of that compound in the CCV standard. In addition, the internal standard for that compound should also be within 0.5 minute of its counterpart in the CCV standard. To confirm the presence of that compound in the sample extract, the mass spectrum of the peak must be evaluated. Spectra are compared against standard spectra of each compound generated on the instrument used for analysis.

4.9.3.2 The following criteria are used to evaluate mass spectra.

4.9.3.2.1 The intensities of the characteristic ions of a compound maximize within one scan of each other. Searches performed based on the presence of a target compound at a compound-specific retention time will be accepted as meeting this criterion.

4.9.3.2.2 All ions present in the standard mass spectra at a relative intensity greater than 30% (most abundant ion in the spectrum equals 100%) or the three ions of greatest intensity must be present in the sample spectrum.

4.9.3.2.3 The relative intensities of ions specified must agree within 30% between the standard and sample spectra. Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample ion abundance must be between 20 and 80 percent.

4.9.3.2.4 If a compound cannot be verified by all of these criteria, but in the technical judgment of the analyst, the identification is correct, then report that identification and proceed with quantification.

4.9.3.2.5 The experienced analyst's judgment weighs heavily in evaluating chromatograms and mass spectra for compound identification. For instance, the retention times of surrogate compounds may be outside their expected windows due to sample matrix effects. If this has occurred, it must be fully documented in the appropriate report notes.

4.9.4 Common Analytical Problems

4.9.4.1 An analyst's professional judgment plays a large role in how data is interpreted. The following guidelines should be followed in order to facilitate consistency between analysts. Any anomalies not addressed in this SOP must be discussed with the supervisor prior to implementation. All anomalies and corrective actions must be documented.

4.9.5 Carryover

4.9.5.1 In some cases, if analytes in a sample are very high, there may be carryover from one analysis to the next. If a sample is suspected of being high due to historical data or extract color, the sample should be diluted prior to analysis, or one or more blanks should be analyzed after the sample to insure that there is no carryover between analyses.

4.9.5.2 However, in the case where high levels were not expected, and do appear in a sample analysis, the analysis after it should be examined carefully for carryover. Any sample analyte whose concentration exceeds 250 ng/µl in the extract should be considered a candidate for carryover.

4.9.5.3 If the following sample has no detected amount of the analyte or analytes which exceeded 250 ng/µl in the previous sample analysis, the following sample does not need to be re-analyzed.

4.9.5.4 If the following sample has detected amounts of the analyte or analytes which exceeded the 250 ng/µl in the previous sample analysis, the sample must be re-analyzed to test for possible carryover.

4.9.6 Manual Peak Integrations

4.9.6.1 Manual peak integrations should be used only when necessary to correct for matrix interference, tangent peaks, and rising baselines. Manual integrations are not to be used in an attempt to meet calibration, surrogate recovery, or spike recovery criteria.

4.9.6.2 If the chromatogram shows degradation due to sample loading e.g. split peaks, lift off, or severe tailing, the sample should be diluted and reanalyzed if required detection limits permit.

4.9.6.3 If a manual integration is necessary, use the following guidelines:

- Integrate only the peak. Start where the peak lifts from the baseline and end as soon as it returns to the baseline. Do not integrate extra baseline in an attempt to increase peak area.
- Do not "peak shave". Do not cut off legitimate parts of the peak in order to reduce peak area.
- In cases of tangent skims, do not increase or decrease peak areas or heights by skimming extra long baselines or drawing the baseline too low.
- Always initial and date your manual integrations.

4.9.7 Compound Quantification

4.9.7.1 Target compound concentrations are calculated using the following equations:

4.9.7.2 Aqueous samples

4.9.7.2.1 The equation for internal standard calculations is

$$Extract\ Concentration(ng / \mu L) = \frac{A_x \times C_{is}}{A_{is} \times RF_{average}}$$

where:

A_x	= Response for the target analyte
A_{is}	= Response for the internal standard
C_{is}	= Concentration of internal standard, in ng/µL
$RF_{average}$	= Average Response Factor (calculated from the initial calibration)

4.9.7.2.2 If the %RSD of a compound's relative response factor is 15% or less, than the concentration may be determined using the average response factor from the initial calibration. Alternatively, the RF may be used as long as the average RSD of all calibration analytes is ≤15%.

4.9.7.2.3 The above equation is used directly by the HP computer to yield the extract concentration.

4.9.7.2.4 To calculate the actual sample concentration, the following calculation must be used.

$$Sample\ Concentration(\mu g / L) = \frac{F \times D \times V_f \times GPC}{V_i}$$

where:

F	= Amount found from HP quantitation report (ng/µL)
D	= Dilution factor of extract
V_f	= Final extract volume (µL)
GPC	= GPC dilution factor. Use 1 if GPC was not performed, 2 if GPC was used
V_i	= Initial sample volume (mL)

4.9.7.2.5 Normally, these calculations are automatically performed by the LIM system.

4.9.7.2.6 Any sample extracts which exceed the upper calibration range for any analyte of interest must be diluted and reanalyzed to bring the analyte into the working range of the calibration.

4.9.7.3 Non-aqueous samples

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

$$Sample\ Concentration(\mu g / L) = \frac{F \times D \times V_f \times GPC}{W_s \times T_s}$$

where:

W_s = Sample size extracted in grams.
T_s = Total Solids in decimal format (i.e. 0.76 not 76).

4.9.7.3.2 Any sample extracts which exceed the upper calibration range for any analyte of interest must be diluted and reanalyzed to bring the analyte into the working range of the calibration.

5. Reports

5.1 Data Packet Organization

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

5.2 Quality Control Reports

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

5.3 Sample Result Reports

5.3.1 Data Qualifying Flags

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

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

B: The analyte of interest was detected in the method blank associated with the sample, as well as in the sample itself. The B flag is applied without regard to the relative concentrations detected in the blank and sample.

J: The analyte of interest was detected below the practical quantitation limit. This value should be regarded as an estimate.

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

X: Indicates an unresolvable isomeric pair. This flag indicates that calculated results are the sum of the two isomers.

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

6. Quality Control

6.1 General Issues

6.1.1 See Appendix VII and VIII for QA/QC limit and corrective action tables.

6.2 Initial Calibration

6.2.1 Criteria

6.2.1.1 Initial calibration data are evaluated using %RSD of the relative response factors as well as minimum average RRFs. The %RSD must be < 30% for each individual Calibration Check Compound (CCC). All other RRFs must be ≤15%. Alternatively, RRFs may be used as long as the average RRFs for all compounds in the method are ≤15% and the CCCs are <30%.

6.2.1.2 Calculate the %RSD for all compounds. CCC criteria are listed in Appendix IV.

6.2.1.3 Calculate the individual and average RFs for each compound.

6.2.1.4 RFs are calculated using the equation

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

where:

A_s = Response of target analyte
A_{is} = Response of internal standard
C_s = Amount of target analyte (ng/µL)
C_{is} = Amount of internal standard (ng/µL)

6.2.1.5 The minimum acceptable average RF for all analytes is 0.050. See Appendix IV for a listing of all SPCCs.

6.2.2 Corrective action

6.2.2.1 If the criteria are not met, other standards may be analyzed or appropriate instrument maintenance and analysis of new standards must be performed. Failure to achieve the required minimum average RRF for the SPCC compounds may indicate the need to perform instrument maintenance or prepare fresh calibration standards.

6.2.2.2 Some compounds, including but not limited to the nitroanilines, the nitrophenols and some additional Appendix IX analytes, tend to show poor or erratic response at the lower calibration levels.

6.2.2.2.1 For routine analyte lists the lowest standard may be omitted from the initial calibration as long as the %RSD does not exceed 30% for any which are CCCs. Additionally, those compounds which are omitted from the lower calibration levels will also have an elevated reporting level to that of the lowest standard.

6.2.2.2.2 For the Appendix IX analytes which exhibit odd calibration behavior refer to Appendix XII for details on calibration.

6.3 Continuing Calibration Verification

6.3.1 Criteria

6.3.1.1 At the beginning of each 12 hour QC period, a CCV standard is analyzed. The RF for each compound is calculated and the percent difference is calculated as follows:

$$\%Difference = \frac{RF_c - RF_i}{RF_i} \times 100$$

where:

RF_i = Average RF from Initial Calibration.
RF_c = RRF from CCV standard.

6.3.1.2 The %D results for all CCCs must be less than 20%. The %D for any other analytes should be < 40% for Navy work.

6.3.1.3 In addition, all compounds must have a minimum RRF of at least 0.050. See Appendix V for all CCV criteria.

6.3.2 Corrective action

6.3.2.1 Check that all peaks have been properly integrated. Check the calculations. If the %D criteria are still not met, perform corrective instrument maintenance or re-tuning, and reanalyze the standard. If the %D or minimum RF criteria are still not met, a new CCV standard may need to be prepared, a new initial calibration performed, a new column installed, or other instrument maintenance performed in order to meet the CCV criteria.

6.4 Method Blank

6.4.1 Criteria

6.4.1.1 A method blank is used to verify contamination-free reagents and apparatus. A method blank is prepared with every set of samples extracted at the same time, at a frequency of at least one blank per 20 samples.

6.4.1.2 The concentration in a method blank of any analyte of concern should not be higher than the report limit. However, since phthalate compounds are common laboratory contaminants, these compounds are allowed to be present in a method blank at up to 5X the RL before corrective action is required. Any analyte response above the MDL is

reported. Values below the reporting limit are "J" flagged. Method blank control limits are detailed in Appendix VI.

6.4.1.3 Where contractually required, e.g. Navy work, all analyte concentrations in the method blank must be <MDL.

6.4.2 Corrective action

6.4.2.1 The blank should be reanalyzed first if carry-over from a previously analyzed sample is believed to be the cause of the contamination. If the contamination is not present in the second analysis, the results of the second analysis may be used. Any similarly affected samples should also be reanalyzed.

6.4.2.2 If an analyte is found in the blank but not in any of the associated samples, the sample batch may not require re-extraction unless this is required by contractual obligations. Consult the QC officer to determine if re-extraction and reanalysis are required.

6.4.2.3 If any analyte exceeds the control limit in the blank and is also found in the associated sample extracts, the samples must be re-extracted and reanalyzed.

6.4.2.4 In any event, it is the laboratory's responsibility to ensure that method interferences caused by contaminants in solvents, reagents, glassware, and other sample processing hardware leading to discrete artifacts and/or elevated baselines in the chromatograms be minimized. In the extreme case of chronic contamination, blanks may have to be analyzed from each stage of the sample processing to determine the contamination source so it can be eliminated. In all cases where blank contamination exceeds the control limit, a narrative comment must be made which documents the corrective actions taken.

6.5 QC Check Sample (LCS)

6.5.1 Criteria

6.5.1.1 The LCS is used to determine whether a method is in control during sample preparation and analysis. A LCS follows the same protocol as the matrix spike analysis except that the spiking solution is added to a blank matrix (deionized water for water or sea sand for soil/sediments) instead of an actual sample.

6.5.2 Corrective action

6.5.2.1 Check all peak integrations and sample calculations.

6.5.2.2 If the analyte recoveries still exceed the control limits, the blank spike extract should be reanalyzed.

6.5.2.3 If the analyte recoveries are still out of control, re-extraction of the associated samples is required.

6.6 Matrix Spike

6.6.1 Criteria

6.6.1.1 A sample is chosen from the samples to be analyzed, and an aliquot of spiking solution is added to this sample prior to extraction. It is not required that a matrix spike analysis be performed with each extraction batch unless the project QAPP requires it. However, the minimum frequency for MS analysis is 1 each per 20 samples per matrix. This matrix spike sample is used to evaluate the matrix effect of the sample upon recovery of selected target analytes.

6.6.1.2 Analyte recovery is calculated as follows:

$$\%Recovery = \frac{SSR - SR}{SA} \times 100$$

where:

SSR = Concentration in spiked sample.
SR = Native concentration in unspiked sample.
SA = Concentration of spike added.

6.6.1.3 MS recovery control limits are empirically derived from Lauck's data and can be found in the Laucks QC Limits database, QC_DB.

6.6.2 Corrective action

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

6.6.2.2 If any analyte recovery exceeds the control limits the following action must be taken:

6.6.2.2.1 If the blank spike analysis yielded recoveries outside the control limits then re-extract and reanalyze all samples extracted with the MS/MSD

6.6.2.2.2 If the blank spike analysis was in control then recoveries outside the control limits can be attributed to matrix effect and sample results are processed as normal. The report comments or narrative section must include comments about the out-of-control recoveries.

6.7 Matrix Spike Duplicate

6.7.1 Criteria

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

6.7.1.2 The calculation for RPD is as follows:

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

where:

S1 = Measured concentration for MS sample.
S2 = Measured concentration for MSD sample.

RPD control limits are detailed in Laucks QC Control Limits database, QC_DB.

6.7.2 Corrective action

6.7.2.1 Corrective action for RPD values which exceed the control limits follows the corrective action for MS/MSD recoveries. If more than one RPD exceeds the control limit, re-extraction may not be required if it can be demonstrated that the sample is non-homogeneous and all MS/MSD recoveries are within the control limits.

6.8 Surrogate Recovery

6.8.1 Criteria

6.8.1.1 Surrogates are chemically similar compounds added to every sample, method blank and QC sample prior to sample processing. They are used to monitor for potential sample processing errors and matrix effects.

6.8.1.2 Surrogate compound recoveries are calculated as follow:

$$\%Recovery = \frac{S_m}{S_a} \times 100$$

where:

S_m = Concentration of surrogate measured in extract.
S_a = Concentration of surrogate added.

6.8.1.3 Surrogate recovery control limits are tabulated in the Laucks QC Control Limits database, NEW_TEST.

6.8.2 Corrective Action

6.8.2.1 Check calculations for possible error.

6.8.2.2 Check instrument performance, if necessary correct the problem and re-analyze the extract.

6.8.2.3 Some samples may require dilution in order to bring one or more target analytes within the calibration range or to overcome significant matrix interference. This may result in the dilution of the surrogate response to the point that the recoveries can not be measured. If the surrogate recoveries are available from a less-diluted or undiluted aliquot of the sample or sample extract, those recoveries may be used to demonstrate that the surrogates were within the QC limits, and no further action is required. For all package work both the diluted and undiluted analyses will be provided.

6.8.2.4 Re-extraction is not necessary in the case where a sample is chosen for MS/MSD analyses and the same recovery pattern is present in all three analyses. In this case, matrix effects may be assumed and re-extraction is not required for this sample.

6.8.2.5 If only one fraction is being analyzed for and surrogates that relate to the other fraction are out-of-control, then re-extraction and reanalysis for the target fraction is not required.

6.8.2.6 All other circumstances require re-extraction and reanalysis of the affected sample(s).

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

7. References

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Method 8270C, "Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique," Revision 2, December 1996, U.S. EPA.

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, 2nd Update, Method 3640A, "Gel-Permeation Cleanup," Revision 1, November 1992, US EPA.

US EPA. Contract Laboratory Program, Statement of Work for Organic Analysis, Multi-Media, Multi-Concentration, Document Number OLM03.1, August 1994.

APPENDIX I

Calibration Standard Solution Concentrations, ng/µL

Compound	STD1	STD2	STD3	STD4	STD5	STD6	Std. Mix#
Phenol	5	10	25	40	60	80	(6)
bis(2-Chloroethyl)ether	5	10	25	40	60	80	(1)
2-Chlorophenol	5	10	25	40	60	80	(6)
1,3-Dichlorobenzene	5	10	25	40	60	80	(2)
1,4-Dichlorobenzene	5	10	25	40	60	80	(2)
Benzyl Alcohol	5	10	25	40	60	80	(5)
1,2-Dichlorobenzene	5	10	25	40	60	80	(2)
2-Methylphenol	5	10	25	40	60	80	(4)
Bis(2-chloroisopropyl)ether	5	10	25	40	60	80	(1)
4-Methylphenol	5	10	25	40	60	80	(6)
N-Nitroso-di-n-propylamine	5	10	25	40	60	80	(1)
Hexachloroethane	5	10	25	40	60	80	(2)
Nitrobenzene	5	10	25	40	60	80	(2)
Isophorone	5	10	25	40	60	80	(2)
2-Nitrophenol	5	10	25	40	60	80	(6)
2,4-Dimethylphenol	5	10	25	40	60	80	(6)
Benzoic Acid	5	10	25	40	60	80	(4)
bis(2-Chloroethoxy)methane	5	10	25	40	60	80	(1)
2,4-Dichlorophenol	5	10	25	40	60	80	(6)
1,2,4-Trichlorobenzene	5	10	25	40	60	80	(2)
Naphthalene	5	10	25	40	60	80	(7)
4-Chloroaniline	5	10	25	40	60	80	(5)
Hexachlorobutadiene	5	10	25	40	60	80	(2)
4-Chloro-3-methylphenol	5	10	25	40	60	80	(6)
2-Methylnaphthalene	5	10	25	40	60	80	(5)
Hexachlorocyclopentadiene	5	10	25	40	60	80	(2)
2,4,6-Trichlorophenol	5	10	25	40	60	80	(6)
2,4,5-Trichlorophenol	5	10	25	40	60	80	(4)
2-Chloronaphthalene	5	10	25	40	60	80	(2)
2-Nitroaniline	5	10	25	40	60	80	(5)
Dimethylphthalate	5	10	25	40	60	80	(1)
Acenaphthylene	5	10	25	40	60	80	(7)
2,6-Dinitrotoluene	5	10	25	40	60	80	(2)
3-Nitroaniline	5	10	25	40	60	80	(5)
Acenaphthene	5	10	25	40	60	80	(7)
2,4-Dinitrophenol	5	10	25	40	60	80	(6)

Compound	STD1	STD2	STD3	STD4	STD5	STD6	Std. Mix#
4-Nitrophenol	5	10	25	40	60	80	(6)
Dibenzofuran	5	10	25	40	60	80	(5)
2,4-Dinitrotoluene	5	10	25	40	60	80	(2)
Diethylphthalate	5	10	25	40	60	80	(1)
4-Chlorophenyl-phenylether	5	10	25	40	60	80	(1)
Fluorene	5	10	25	40	60	80	(7)
4-Nitroaniline	5	10	25	40	60	80	(5)
4,6-Dinitro-2-methylphenol	5	10	25	40	60	80	(6)
N-Nitrosodiphenylamine	5	10	25	40	60	80	(1)
4-Bromophenyl-phenylether	5	10	25	40	60	80	(1)
Hexachlorobenzene	5	10	25	40	60	80	(2)
Pentachlorophenol	5	10	25	40	60	80	(6)
Phenanthrene	5	10	25	40	60	80	(7)
Anthracene	5	10	25	40	60	80	(7)
Carbazole	5	10	25	40	60	80	(2)
Di-n-butylphthalate	5	10	25	40	60	80	(1)
Fluoranthene	5	10	25	40	60	80	(7)
Pyrene	5	10	25	40	60	80	(7)
Butylbenzylphthalate	5	10	25	40	60	80	(1)
3,3'-Dichlorobenzidine	5	10	25	40	60	80	(3)
Benzo(a)anthracene	5	10	25	40	60	80	(7)
Chrysene	5	10	25	40	60	80	(7)
bis(2-Ethylhexyl)phthalate	5	10	25	40	60	80	(1)
Di-n-octylphthalate	5	10	25	40	60	80	(1)
Benzo(b)fluoranthene	5	10	25	40	60	80	(7)
Benzo(k)fluoranthene	5	10	25	40	60	80	(7)
Benzo(a)pyrene	5	10	25	40	60	80	(7)
Indeno(1,2,3-cd)pyrene	5	10	25	40	60	80	(7)
Dibenzo(a,h)anthracene	5	10	25	40	60	80	(7)
Benzo(g,h,i)perylene	5	10	25	40	60	80	(7)
N-Nitrosodimethylamine	5	10	25	40	60	80	(1)
2-Fluorophenol	5	10	25	40	60	80	(8)
Aniline	5	10	25	40	60	80	(5)
Phenol-d5	5	10	25	40	60	80	(8)
2-Chlorophenol-d4	5	10	25	40	60	80	(8)
1,2-Dichlorobenzene-d4	5	10	25	40	60	80	(9)
Nitrobenzene-d5	5	10	25	40	60	80	(9)
2-Fluorobiphenyl	5	10	25	40	60	80	(9)
2,4,6-Tribromophenol	5	10	25	40	60	80	(8)

Compound	STD1	STD2	STD3	STD4	STD5	STD6	Std. Mix#
1,2-Diphenylhydrazine	5	10	25	40	60	80	(2)
Benzidine	5	10	25	40	60	80	(3)
Terphenyl-d14	5	10	25	40	60	80	(9)

APPENDIX II

DFTPP Tuning Criteria

Mass Relative Abundance

51	30-80% of mass 198
68	less than 2% of 69
69	present
70	less than 2% of 69
127	25-75% of mass 198
197	less than 1% of 198
198	100%
199	5-9% of mass 198
275	10-30% of mass 198
365	greater than 0.75% of mass 198
441	0-100 % of mass 443
442	40-110% of mass 198
443	15-24% of mass 442

CLP Criteria has been used as allowed by the method. The spectrum must be taken by averaging the peak apex with each of the adjacent scans and background subtracting not greater than 20 scans prior to the beginning of DFTPP.

APPENDIX III

Data Packet Order List

QC Summary
Surrogate Recovery Summary Report
Blank Spike Recovery Report
MS/MSD Recovery Report
Method Blank Summary
GC/MS Instrument Performance Check
Internal Standard Area and RT Summary Report

Sample Data
Target Compound Results (Organics Analysis Data Sheet)
Tentatively Identified Compound (TIC) Results (if required)
Sample Chromatograms, quantitation reports and spectra for all samples

Standards Data
Initial Calibration Summary Report
Chromatograms and quantitation reports for all initial calibration standards
Continuing Calibration Summary Reports
Chromatograms and quantitation reports for all CCV Standards

Raw QC Data
Bar Graph Spectrum, mass listing and chromatogram for every DFTPP injection
Method Blank Data
Target Compound Results (Organics Analysis Data Sheet)
Tentatively Identified Compound (TIC) Results (if required)
Chromatograms, quantitation reports and spectra for all method blanks
Blank Spike Data
Target Compound Results (Organics Analysis Data Sheet)
Chromatograms, quantitation reports and spectra for all blank spikes
Matrix Spike/Matrix Spike Duplicate Data
Target Compound Results (Organics Analysis Data Sheet)
Chromatograms, quantitation reports and spectra for all MS analyses
Control Charts

Bench Sheets
All extraction bench sheets, analysis log book pages, chains of custody, and any other pertinent information

APPENDIX IV

Initial Calibration Criteria

Calibration Check Compound	%RSD Limit
Phenol	< 30%
1,4-Dichlorobenzene	< 30%
2-Nitrophenol	< 30%
2,4-Dichlorophenol	< 30%
Hexachlorobutadiene	< 30%
4-Chloro-3-methylphenol	< 30%
2,4,6-Trichlorophenol	< 30%
Acenaphthene	< 30%
N-Nitrosodiphenylamine	< 30%
Pentachlorophenol	< 30%
Fluoranthene	< 30%
Di-n-octylphthalate	< 30%
Benzo(a)pyrene	< 30%

System Performance Check Compound	Minimum Average RF
N-Nitroso-di-n-propylamine	0.050
Hexachlorocyclopentadiene	0.050
2,4-Dinitrophenol	0.050
4-Nitrophenol	0.050

Note: All analytes must have RFs 0.050.

APPENDIX V

Continuing Calibration Verification Criteria

Calibration Check Compound	%D Limit
Phenol	20%
1,4-Dichlorobenzene	20%
2-Nitrophenol	20%
2,4-Dichlorophenol	20%
Hexachlorobutadiene	20%
4-Chloro-3-methylphenol	20%
2,4,6-Trichlorophenol	20%
Acenaphthene	20%
N-Nitrosodiphenylamine	20%
Pentachlorophenol	20%
Fluoranthene	20%
Di-n-octylphthalate	20%
Benzo(a)pyrene	20%

System Performance Check Compound	Minimum RF
N-Nitroso-di-n-propylamine	0.050
Hexachlorocyclopentadiene	0.050
2,4-Dinitrophenol	0.050
4-Nitrophenol	0.050

Note: All analytes must have RFs 0.050.

APPENDIX VI

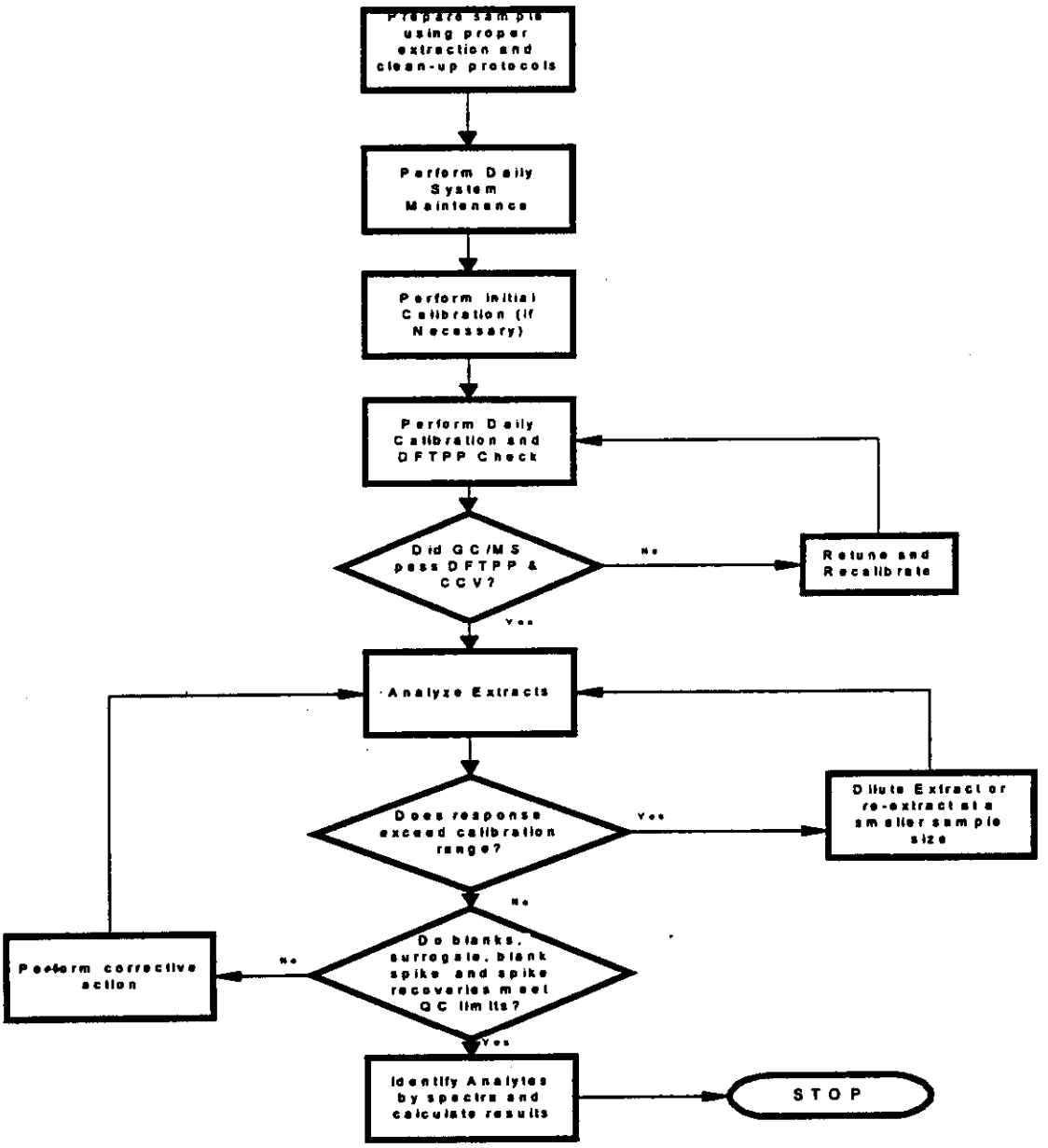
Method Blank Control Limits

Compound	Control Limits Water	Soil
Phthalates	5X RL	5X RL
All other compounds	RL	RL

If contractually required, e.g. Navy work, all analyte concentrations must be < MDL in the method blank.

APPENDIX VII

Method 8270 Flow Chart



APPENDIX VIII

Method 8270C QA Requirements and Corrective Actions

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
DFTPP Tuning Verification (50 ng)	Mass Abundance 51 30-80% of 198 68 <2% of 69 69 present 70 <2% of 69 127 25-75% of 198 197 <1% of 198 198 100% 199 5-9% of 198 275 10-30% of 198 365 >.75% of 198 441 present but < 443 442 40-110% of 198 443 15-24% of 442	Mass Abundance 51 30-80% of 198 68 <2% of 69 69 present 70 <2% of 69 127 25-75% of 198 197 <1% of 198 198 100% 199 5-9% of 198 275 10-30% of 198 365 >.75% of 198 441 present but < 443 442 40-110% of 198 443 15-24% of 442	Every 12 hours; CLP Criteria is used as allowed by the method.	Retune instrument to pass criteria.	
Initial Calibration	Minimum of 5 levels, lowest near but above MDL, %RSD for CCC <30, All others should be <15%. RF for SPCC >0.05.	6 levels, %RSD for CCC <30, All others <15%, All RFs >0.05 Alternatively, average RSD must be <15%.	At minimum, yearly or as necessary due to major instrument maintenance or continuing difficulties meeting the CCV requirements.	Re-analysis of out of control standards.	Copies of all raw data, mass calibration, tune, and Form VI.
Continuing Calibration Verification	Mid-level standard every 12 hours. %D <20% for CCCs SPCCs >0.05. IS Area must be -50% to +100% of the IS in the ICV. RT of IS must be within ±30 seconds of the RT of the IS in the IC.	25 ng/µl standard every 12 hours. %D for CCC<20. All RFs >0.05 Average %D must be <15%. IS Area must be -50% to +100% of the IS in the IC. No RT limits for IS in the CCV.	Every 12 hours.	Perform system maintenance, re-analyze CCV standard.	
Internal Standards	1,4-Dichlorobenzene-d4, Naphthalene-d8, Acenaphthene-d10, Phenanthrene-d10, Chrysene-d12, Perylene-d12, all @ 40 ng/uL ea..	1,4-Dichlorobenzene-d4, Naphthalene-d8, Acenaphthene-d10, Phenanthrene-d10, Chrysene-d12, Perylene-d12, all @ 20 ng/µl ea. Area must be -50% to + 100% of the area of the IS in the CCV. RT must be within 0.5 minutes of RT in CCV.	All standards, blanks, matrix spikes, matrix spike duplicates, blank spikes, SRMs, and sample re-extracts.	Re-analyze any sample which IS areas or RTs are out of range.	
Method Blank	Analytes must be <MDL except phthalates which must be <5X the MDL.	All analytes <RL. Except phthalates up to 5X RL.	One method blank per 20 samples or each extraction batch of samples, whichever is more frequent.	Re-analyze blank. If still out of control, re-extract the entire batch of samples unless the offending analyte(s) are not detected in the associated samples.	Notation in instrument log and case narrative if applicable.

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Surrogate Recovery	Nitrobenzene-d5, 2-Fluorobiphenyl, Terphenyl-d14, Phenol-d6, 2-Fluorophenol, & 2,4,6-Tribromophenol. Upper and lower recovery limits determined by 3X standard deviation of at least twenty samples. To be performed at least semi-annually.	Nitrobenzene-d5, 2-Fluorobiphenyl, Terphenyl-d14, Phenol-d6, 2-Fluorophenol, & 2,4,6-Tribromophenol. Upper and lower recovery limits determined by 3X standard deviation of at least twenty samples. To be performed at least semi-annually.	All samples, method blanks, LCSs, LESs.	Re-extraction of the sample is required if any surrogate is <10% recovery or 2 acid surrogates or 2 B/N surrogates are out of control. All surrogates must be in control in the method blank, otherwise all associated samples must be re-extracted. Where contractually required, e.g. Navy work, all surrogates must be within control limits.	Any out of control surrogates are to be documented in the instrument logbook and the job comments section or case narrative.
Blank Spike Recovery	MS/MSD Recovery criteria	Empirically derived from five or more blank spikes. QC limit equals 3X.	One per batch of twenty.	Re-extract batch unless MS/MSD is perfect.	Narrate in case narrative.
MS/MSD Recovery & RPD	Empirically derived from five or more MS/MSD pairs. QC interval equals 3X standard deviation.	Empirically derived from five or more MS/MSD pairs. QC interval equals 3X standard deviation.	One per batch of twenty.	Re-extract batch unless blanks spike is perfect or documentable matrix effect is present.	Narrate problems in case narrative.

APPENDIX IX

Appendix IX Calibration Standard Solution Concentrations, ng/µL

Compound	STD1	STD2	STD3	STD4	STD5	Std Mix #
2,6-Dichlorophenol	10	25	40	60	80	(A5)
1,2,4,5-Tetrachlorobenzene	10	25	40	60	80	(A2)
2,3,4,6-Tetrachlorophenol	10	25	40	60	80	(A5)
Dinoseb	10	25	40	60	80	(A5)
Methanesulfonate, methyl	10	25	40	60	80	(A6)
Methanesulfonate, ethyl	10	25	40	60	80	(A6)
Phosphorothiotic Acid	10	25	40	60	80	(A4)
Safrole	10	25	40	60	80	(A6)
Isosafrole, cis & trans	10	25	40	60	80	(A6)
1,4-Naphthoquinone	10	25	40	60	80	(A6)
1,3-Dinitrobenzene	10	25	40	60	80	(A6)
Thionazin	10	25	40	60	80	(A4)
2,4-D, methyl ester	10	25	40	60	80	(A4)
Diallate	10	25	40	60	80	(A4)
Phorate	10	25	40	60	80	(A4)
Dimethoate	10	25	40	60	80	(A4)
Silvex, methyl ester	10	25	40	60	80	(A4)
2,4,5-T, methyl ester	10	25	40	60	80	(A4)
Pronamide	10	25	40	60	80	(A6)
Disulfoton	10	25	40	60	80	(A4)
Methyl parathion	10	25	40	60	80	(A4)
Ethyl parathion	10	25	40	60	80	(A4)
Isodrin	10	25	40	60	80	(A6)
Chlorobenzilate	10	25	40	60	80	(A6)
Kepone	10	25	40	60	80	(A6)
Famphur	10	25	40	60	80	(A4)
3-Methylcholanthrene	10	25	40	60	80	(A6)
Ethanamine,N-methyl-N-nitroso	10	25	40	60	80	(A3)
N-nitrosomorpholine	10	25	40	60	80	(A3)
Butanamine, N-butyl-N-nitroso	10	25	40	60	80	(A3)
2-Naphthalenamine	10	25	40	60	80	(A3)
Diphenylamine	10	25	40	60	80	(A3)
1,3,5-Trinitrobenzene	10	25	40	60	80	(A3)
4-Aminobiphenyl	10	25	40	60	80	(A3)
Methyapyrilene, HCL	10	25	40	60	80	(A3)

Compound	STD1	STD2	STD3	STD4	STD5	Std Mix #
p-Dimethylaminoazobenzene	10	25	40	60	80	(A3)
2-Acetylaminofluorene	10	25	40	60	80	(A3)
7,12-Dimethylbenz(a)anthracene	10	25	40	60	80	(A3)
2-Picoline	10	25	40	60	80	(A7)
Pentachloroethane	10	25	40	60	80	(A7)
Acetophenone	10	25	40	60	80	(A7)
N-Nitrosopyrrolidine	10	25	40	60	80	(A7)
N-Nitrosopiperidine	10	25	40	60	80	(A7)
Hexachloropropene	10	25	40	60	80	(A7)
1,4-Phenylenediamine	10	25	40	60	80	(A7)
Pentachlorobenzene	10	25	40	60	80	(A7)
1-Naphthylamine	10	25	40	60	80	(A7)
5-Nitro-o-toluidine	10	25	40	60	80	(A7)
Pentachloronitrobenzene	10	25	40	60	80	(A7)
N-Nitroquinoline-N-oxide	10	25	40	60	80	(A7)
3,3-Dimethylbenzidine	10	25	40	60	80	(A7)
Hexachlorophene	10	25	40	60	80	(A7)
N-Nitrosodiethylamine	10	25	40	60	80	(A7)
o-Toluidine	10	25	40	60	80	(A7)
Phentermine	10	25	40	60	80	(A7)
Sulfotep	10	25	40	60	80	(A7)
Aramite	10	25	40	60	80	(A7)
Phenacetin	10	25	40	60	80	(A7)

Appendix X

Analytes Amenable to Analysis by Method 8270C

Compound	Current 8270 Target Analytes: Primary Ion	Secondary Ion(s)	Retention Time (min)
Phenol	94	65, 66	12.20
bis(2-Chloroethyl)ether	93	63, 95	12.32
2-Chlorophenol	128	64, 130	12.33
1,3-Dichlorobenzene	146	148, 111	12.69
1,4-Dichlorobenzene	146	148, 111	12.87
Benzyl Alcohol	108	79, 77	13.44
1,2-Dichlorobenzene	146	148, 111	13.39
2-Methylphenol	107	108, 77	13.86
Bis(2-chloroisopropyl)ether	45	77, 121	13.88
4-Methylphenol	107	108, 77	14.33
N-Nitroso-di-n-propylamine	70	42, 101	14.39
Hexachloroethane	117	201, 199	14.27
Nitrobenzene	77	123, 65	14.64
Isophorone	82	195, 138	15.42
2-Nitrophenol	139	109, 65	15.59
2,4-Dimethylphenol	122	107, 121	15.89
Benzoic Acid	122	105, 77	16.62
bis(2-Chloroethoxy)methane	93	95, 123	16.16
2,4-Dichlorophenol	162	164, 98	16.34
1,2,4-Trichlorobenzene	180	182, 145	16.55
Naphthalene	128	129, 127	16.71
4-Chloroaniline	127	129, 65	17.04
Hexachlorobutadiene	225	223, 227	17.32
4-Chloro-3-methylphenol	107	144, 142	18.65
2-Methylnaphthalene	142	141	18.87
Hexachlorocyclopentadiene	237	235, 272	19.61
2,4,6-Trichlorophenol	196	198, 200	19.91
2,4,5-Trichlorophenol	196	198, 97	20.02
2-Chloronaphthalene	162	127, 164	20.39
2-Nitroaniline	65	92, 138	20.92
Dimethylphthalate	163	194, 164	21.68
Acenaphthylene	152	151, 153	21.71
2,6-Dinitrotoluene	165	63, 89	21.88

Current 8270 Target Analytes:

Compound	Primary Ion	Secondary Ion(s)	Retention Time (min)
3-Nitroaniline	138	108, 92	20.92
Acenaphthene	154	153, 152	22.32
2,4-Dinitrophenol	184	63, 154	22.59
4-Nitrophenol	139	109, 65	22.92
Dibenzofuran	168	139	22.83
2,4-Dinitrotoluene	167		23.08
4-Nitroaniline	138	65, 108	24.27
4,6-Dinitro-2-methylphenol	198	51, 105	24.39
N-Nitrosodiphenylamine	169	168, 167	24.45
4-Bromophenyl-phenylether	248	250, 141	25.51
4-Chlorophenyl-phenylether	204	206, 141	23.99
Fluorene	166	165, 167	23.92
Diethylphthalate	149	177, 150	23.95
Hexachlorobenzene	284	142, 249	25.93
Pentachlorophenol	266	264, 268	26.54
Phenanthrene	178	179, 176	26.94
Anthracene	178	176, 179	27.07
Carbazole	167	84	27.67
Di-n-butylphthalate	149	150	29.10
Fluoranthene	202	101	30.74
Pyrene	202	200, 203	31.42
Butylbenzylphthalate	149	91	33.82
3,3'-Dichlorobenzidine	252	254, 126	35.33
Benzo(a)anthracene	228	229, 226	35.27
Chrysene	228	226, 229	35.41
bis(2-Ethylhexyl)phthalate	149	167, 279	35.74
Di-n-octylphthalate	149	167, 43	37.57
Benzo(b)fluoranthene	252	253, 125	38.54
Benzo(k)fluoranthene	252	253, 125	38.63
Benzo(a)pyrene	252	253, 125	39.55
Indeno(1,2,3-cd)pyrene	276	138, 227	43.87
Dibenzo(a,h)anthracene	278	139, 279	43.99
Benzo(g,h,i)perylene	276	138, 277	45.12
N-Nitrosodimethylamine	74	42	5.76
2-Fluorophenol	112	64	9.46
Aniline	93	66, 65	12.08

Current 8270 Target Analytes:

Compound	Primary Ion	Secondary Ion(s)	Retention Time (min)
Phenol-d5	99	42, 71	12.17
2-Chlorophenol-d4	132	68	12.29
1,2-Dichlorobenzene-d4	152	115	13.36
Nitrobenzene-d5	82	128, 54	14.58
2-Fluorobiphenyl	172	171	20.16
2,4,6-Tribromophenol	330	332, 141	24.77
1,2-Diphenylhydrazine	77	182, 105	24.48
Benzidine	184	92, 185	31.27
Terphenyl-d14	244	122, 212	32.06

Current Appendix IX Analytes:

Compound	Primary Ion	Secondary Ion(s)	Retention Time (min)
2,6-Dichlorophenol	162	164, 63	16.02
1,2,4,5-Tetrachlorobenzene	216	214, 218	18.48
2,3,4,6-Tetrachlorophenol	230	232, 234	22.32
Dinoseb	211	163, 117	26.11
Methanesulfonate, methyl	80	79, 95	8.27
Methanesulfonate, ethyl	109	97	10.17
Phosphorothiotic Acid	97	121, 198	15.22
Safrole	162	104, 131	17.69
Isosafrole, cis & trans	162	104, 131	19.31
1,4-Naphthoquinone	158	102, 104	19.96
1,3-Dinitrobenzene	168	76, 122	20.63
Thionazin	97	96, 107	23.10
2,4-D, methyl ester	199	175, 234	23.55
Diallate	86	234, 236	24.40
Phorate	260	231, 121	24.44
Dimethoate	87	93, 125	25.04
Silvex, methyl ester	196	225, 282	25.33
2,4,5-T, methyl ester	233	235, 268	25.75
Pronamide	173	175, 255	25.77
Disulfoton	88	274, 142	26.06
Methyl parathion	263	125, 109	27.25
Ethyl parathion	291	235, 186	28.53
Isodrin	193	195, 230	29.07

Current Appendix IX Analytes:

Compound	Primary Ion	Secondary Ion(s)	Retention Time (min)
Chlorobenzilate	139	251, 253	31.64
Kepone	272	274, 355	32.30
Famphur	218	125, 217	32.52
3-Methylcholanthrene	268	252, 253	39.26
Ethanamine,N-methyl-N-nitroso	42	88, 43	7.08
N-nitrosomorpholine	56	86, 116	13.06
Butanamine, N-butyl-N-nitroso	84	41, 57	16.99
2-Naphthalenamine	143	144, 115	21.99
Diphenylamine	169	168, 167	23.07
1,3,5-Trinitrobenzene	213	75, 74	24.23
4-Aminobiphenyl	169	168, 170	25.02
Methyapyrilene, HCL	58	97, 191	28.52
p-Dimethylaminoazobenzene	120	225, 226	31.14
2-Acetylaminofluorene	181	180, 223	33.04
7,12-Dimethylbenz(a)anthracene	256	241, 239	36.97
2-Picoline	93	92, 78	7.70
Pentachloroethane	167	165, 119	12.00
Acetophenone	105	77, 51	14.19
N-Nitrosopyrrolidine	100	41, 42	14.24
N-Nitrosopiperidine	42	114, 55	15.16
Hexachloropropene	213	215, 211	17.12
1,4-Phenylenediamine	108	80, 81	18.19
Pentachlorobenzene	250	252, 248	22.94
1-Naphthylamine	143	144, 115	23.16
5-Nitro-o-toluidine	152	77, 106	24.22
Pentachloronitrobenzene	237	214, 295	26.82
N-Nitroquinoline-N-oxide	190	160, 89	29.60
3,3-Dimethylbenzidine	212	213, 211	33.69
Hexachlorophene	196	198, 209	39.26
N-Nitrosodiethylamine	74	42	9.88
o-Toluidine	106	107	14.05
Phentermine	58	91, 134	15.95
Sulfotep	97	322, 202	25.09
Aramite	135	193, 208	23.29
Phenacetin	108	109, 179	25.39

APPENDIX XI

Semivolatile Internal Standards With Corresponding Analytes Assigned For Quantitation

1,4-Dichlorobenzene-d4	Naphthalene-d8	Acenaphthene-d10
Phenol	Nitrobenzene	Hexachlorocyclopentadiene
Bis(2-Chloroethyl)ether	Isophorone	2,4,6-Trichlorophenol
2-Chlorophenol	2-Nitrophenol	2,4,5-Trichlorophenol
1,3-Dichlorobenzene	2,4-Dimethylphenol	2-Chloronaphthalene
1,4-Dichlorobenzene	Benzoic Acid	2-Nitroaniline
Benzyl Alcohol	bis(2-Chloroethoxy)methane	Dimethylphthalate
1,2-Dichlorobenzene	2,4-Dichlorophenol	Acenaphthylene
2-Methylphenol	1,2,4-Trichlorobenzene	2,6-Dinitrotoluene
Bis(2-chloroisopropyl)ether	Naphthalene	3-Nitroaniline
4-Methylphenol	4-Chloroaniline	Acenaphthene
N-Nitroso-di-n-propylamine	Hexachlorobutadiene	2,4-Dinitrophenol
Hexachloroethane	4-Chloro-3-methylphenol	4-Nitrophenol
Aniline	2-Methylnaphthalene	Dibenzofuran
Pyridine	Nitrobenzene-d5	2,4-Dinitrotoluene
Phenol-d5	2,6-Dichlorophenol	4-Nitroaniline
2-Chlorophenol-d4	Safrole	Fluorene
1,2-Dichlorobenzene-d4	Isosafrole, cis & trans	4-Chlorophenyl-phenylether
N-Nitrosodimethylamine	1,4-Naphthoquinone	Diethylphthalate
2-Fluorophenol	Butanamine, N-methyl-N-nitroso	1,2,4,5-Tetrachlorobenzene
Methanesulfonate methyl	Hexachloropropene	2,3,4,6-Tetrachlorophenol
Methanesulfonate ethyl	1,4-phenylenediamine	1,3-Dinitrobenzene
Phosphorothiotic acid	Phentermine	Thionazin
Ethanamine, N-methyl-N-nitroso		2,4-D methyl ester
N-Nitrosomorpholine		Diallate
2-Picoline		Phorate
Pentachloroethane		Dimethoate
Acetophenone		Silvex methyl ester
N-Nitrosopyrrolidine		2,4,5-T methyl ester
N-Nitrosopiperidine		2-Naphthaleneamine

1,4-Dichlorobenzene-d4	Naphthalene-d8	Acenaphthene-d10
N-Nitrosodiethylamine O-Toluidine		Pentachlorobenzene 1-Naphthylamine 5-Nitro-o-toluidine Pentachloronitrobenzene Sulfotep Aramite

Phenanthrene-d10	Chrysene-d12	Perylene-d12
4,6-Dinitro-2-methylphenol N-Nitrosodiphenylamine 4-Bromophenyl-phenylether Hexachlorobenzene Pentachlorophenol Phenanthrene Anthracene Carbazole Di-n-butylphthalate Fluoranthene 2,4,6-Tribromophenol 1,2-Diphenylhydrazine Dinoseb Pronamide Disulfoton Methyl parathion Ethyl parathion Isodrin Diphenylamine 1,3,5-Trinitrobenzene 4-Aminobiphenyl Methapyrilene, HCL N-Nitroquinoline-n-oxide 3,3-Dimethylbenzidine Phenacetin	Pyrene Butylbenzylphthalate 3,3'-Dichlorobenzidine Benzo(a)anthracene Chrysene bis(2-Ethylhexyl)phthalate Benzidine Terphenyl-d14 Chlorobenzilate Kepone Famphur P-Dimethylaminoazobenzene 2-Acetylaminofluorene 7,12- Dimethylbenz(a)anthracene Hexachlorophene	Di-n-octylphthalate Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene Indeno(1,2,3-cd)pyrene Dibenzo(a,h)anthracene Benzo(g,h,i)perylene 3-Methylcholanthrene

APPENDIX XII

Comments On Analysis Of Appendix IX Semivolatile Compounds

Stock solutions of target Appendix IX compounds listed above were purchased from a commercial source (e.g. Supelco).

Appendix IX Semi-Volatile Mix 2	**# 4-6971**	**(A2)**
Appendix IX Semi-Volatile Mix 3	**# 4-6972**	**(A3)**
Appendix IX Semi-Volatile Mix 4	**# 4-6973**	**(A4)**
Appendix IX Semi-Volatile Mix 5	**# 4-6974**	**(A5)**
Appendix IX Semi-Volatile Mix 6	**# 4-7601**	**(A6)**

Some additional analytes required for analysis were not present in these available solutions. Therefore, a special certified custom mix was ordered from Supelco under lot # LA-69411 (A7). All purchased stock solutions contained all analytes of interest at a concentration of 2000 µg/mL each in methylene chloride.

Calibration standards were prepared in methylene chloride at five concentration levels (10, 25, 40, 60 and 80 ng/µL). Each calibration standard contains all compounds of interest, surrogates, and internal standards. The internal standards were added so that they were present in all calibration standards at a concentration of 20 ng/µL each.

Through preparation of mixed Appendix IX semivolatile standards, it has been documented that aramite, safrole, isosafrole, 3-methylcholanthrene , 3,3-dimethylbenzidine, methapyrilene HCL, hexachlorophene, and alpha,alpha-dimethylphenethylamine are unstable when combined in working solutions with other semivolatile target compounds, which limited expiration of the standard mixes to 3 months only.

Due to the nature of the Appendix IX compounds it was impossible to combine all six standard mixtures into one solution. Instead, Mixes #2 & #3 only were mixed together and the remaining standards were analyzed individually and resulted in good analytical resolution and easy identification of each components. Most of the analytes demonstrated excellent linearity, and their response factors were constant over the calibration range (%RSD <15). However, some analytes like kepone, 1,3,5-trinitrobenzene, 7,12-dimethylbenz(a) anthracene, 1,4-phenylenediamine, n-nitroquinoline-n-oxide, and hexachlorophene performed poorly due to the limited sensitivity and/or erratic chromatographic behavior.

Comments - Continued

Also it was discovered that n-nitrosodiphenylamine decomposes in the injection port and can not be separated with diphenylamine. Diastereoisomeric pair aramite and isosafrole can hardly be resolved on the RTX-5 column. Diallate and phorate as well as the pair of 2,4,5-T methyl ester and pronamide components are not resolved chromatographically and produce mass spectra containing ions contributed by both co-eluting analytes.

For Appendix IX and TCLP analyses, the compound 3-methylphenol (m-Cresol) coelutes with 4-methylphenol (p-Cresol). Since the spectrum and retention time for these two compounds are the same, these two compounds will be reported together as the sum of these two compounds and with the assumption that 3-methylphenol has the same response factor as the 4-methylphenol present in the standards.

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

James Hansen
SIGNATURE

Eng. Prot. Dept. Mgr.
TITLE

5/16/00
DATE