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Work Plan
for
RCRA Facility Investigation, Corrective Measures Study, and Risk Assessment
at
Solid Waste Management Unit 2 - Dye Burial Grounds

Naval Surface Warfare Center Crane Division
Crane, Indiana

Volume II of II - Analytical SOPs

Southern Division Naval Facilities Engineering Command

Contract Number N62467-94-D-0888

Contract Task Order 0010

May 2001

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FOR
RCRA FACILITY INVESTIGATION, CORRECTIVE MEASURES STUDY, AND RISK ASSESSMENT
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NAVAL SURFACE WARFARE CENTER
CRANE, DIVISION
CRANE, INDIANA

VOLUME II OF II - ANALYTICAL SOPs

COMPREHENSIVE LONG-TERM
ENVIRONMENTAL ACTION NAVY (CLEAN) CONTRACT

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ATTACHMENT A

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SOP Number	Revision	Title
Laucks Testing Laboratories, Inc. SOPs:		
• LTL-1001	4	Elements of SOP and Method Formats
• LTL-1002	5	Document Tracking and Control
• LTL-1003	2	Chain-of-Custody and Documentation Procedures
• LTL-1004	4	Documentation of Analyst Competence and Training
• LTL-1005	5	Analytical Balances
• LTL-1006	3	Refrigerator, Freezer, and Oven Thermometer Calibration and Maintenance
• LTL-1007	3	Maintaining Instrument Records and Logbooks
• LTL-1008	6	QC Corrective Action
• LTL-1009	3	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	4	Preparation, Storage, Shelf Life and Traceability Documentation of Standards and Reference Materials
• LTL-1017	1	Internal Audit Procedures
• LTL-1018	1	Overview of Review and Approval Practices for Validatable Data Packages
• LTL-1019	1	Controlling, Maintaining, and Monitoring Laboratory Logbooks
• LTL-2001	5	Waste Segregation and Disposal
• LTL-4002	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-6006	0	Cation Exchange Capcity of Soils (Sodium Acetate) by SW 846 Method 9081
• LTL-7003	2	Inorganic Glass Cleaning Procedure
• LTL-7009	3	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-7012	1	Microwave Assisted Acid Digestion of Sediments, Sludges, Soils, and Oils Using SW 846 Method 3051
• LTL-7015	1	Acid Digestion of Sediments, Sludges, and Soils Using SW 846 Method 3050B
• LTL-7101	3	Operation of the Jarrell-Ash Enviro36 Simultaneous ICP
• LTL-7105	1	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-9104	2	Preparation and Analysis of Total and Amenable Cyanide according to EPA Methods 335.1, 335.3, and SW 846 Method 9012A
• LTL-9110	6	Determination of Anions by Ion Chromatography by Method 300.0A or 9056
• LTL-9113	2	pH in Soil and Waste Samples Using SW 846 Method 9045C
• LTL-9115	2	The Determination of Total Organic Carbon in Water Samples Based on SW846 9060

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- LTL-9202 3 Total Suspended Solids (Non-Filterable Residue) Using EPA Method 160.2/Standard Method 2540D and Total Volatile Suspended Solids Using Standard Methods 2540E

NSWC Crane Laboratory SOPs:

- CR4052-PD-001 0 Document Control and Tracking
- CR4052-PD-002 0 Elements and Format of Procedure Documents
- CR4052-PD-003 0 Internal Sample Chain of Custody
- CR4052-PD-004 0 Glass Cleaning Procedure
- CR4052-PD-005 0 Analytical Balances
- CR4052-PD-006 0 Refrigerator, Freezer and Oven Thermonmeter Calibration, Monitoring and Maintenance
- CR4052-PD-007 0 Waste Segregation and Disposal
- CR4052-PD-008 0 Controlling, Maintaining and Monitoring Instrument Records, Instrument Logbooks and Laboratory Notebooks
- CR4052-PD-009 0 Quality Assurance
- CR4052-PD-011 0 Percent Mositure Analysis in Soil
- CR4052-PD-012 0 Determination of the Density of Soil Containing Organic Colorants by the Drive-Cylinder Method
- CR4052-PD-013 0 Particle Size Analysis of Soil Containing Organic Colorants
- CR4052-PD-2500 1 Determination of Organic Colorants in Environmental Matrices by HPLC
- CR-405-3001 A Directive for Contrtol of Technical Documents and Engineering Drawings
- NAVSURFWARCENDIVCRANE INSTRUCTION 4855.1A Crane Division Quality Manual
- NAVSURFWARCENDIVCRANE INSTRUCTION 5090.2B Hazardous Waste Management Plan
- NAVSURFWARCENDIVCRANE INSTRUCTION 4855.20 Calibration Program
- NAVSURFWARCENDIVCRANE INSTRUCTION 4855.4 Shelf-life Management

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

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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-1002

Title: **Document Tracking and Control**

Revision history:

Number	Date
1.0	05/15/95
2.0	12/27/95
3.0	1/06/98
4.0	12/14/99
5.0	6/8/2000

Written by: Harry Romberg Date: 06-13-2000
Harry Romberg, QA Officer

Approved by: Kathy Kreps Date: 06/13/00
Kathy Kreps, Laboratory Director

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No. 20 Assigned to: Tetra

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APPENDIX E **17**

SOP UPDATE FORM 17

1. Introduction and Scope

1.1 Purpose

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

1.2 Document Types

1.2.1 Laucks recognizes two types of documents.

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

1.3 Scope

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

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

2. Operation Procedures

2.1 Initiation and Updating of Documents

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

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

2.1.3 Copies of this form will be given to the responsible individual and the appropriate Division Manager. Originals will be kept on file in the QA Department. This insures that all responsible parties are informed of the initiation of the creation or revision process. This form should be filled out as soon as it is determined that the creation or revision of a document is necessary and a responsible party has been assigned. These forms will also be issued approximately every other year, depending upon the last review or revision date, in order to initiate the review process for existing SOPs. These forms may also be issued when a significant change is made in a procedure or when required in response to an audit or document review that points out deficiencies that require correction prior to the regularly scheduled review date. The QA Plan will be reviewed and revised approximately annually.

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

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

2.2 Tracking and Control of Existing Documents

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

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

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

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

2.2.4 SOPs, Methods, and many other documents must have header information which clearly indicates the document number, revision, date of revision, and document replaced by revision. Page numbers and the total number of pages in the document are strongly recommended and usually required, although some discretion may be allowed by QA in

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

SOP No: LTL-xxxx
Revision: 1
Date: 06/08/00
Page: x of xx
Replaces: 0

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

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

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

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

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

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

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

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

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

- Newly distributed versions are placed in the SOP manuals.
- The signature lists for the current documents are signed and dated as staff complete reading the SOPs. In addition, as staff new to a particular task (SOP) are trained, the departmental supervisor will ensure that they have read and signed the signature list for that SOP. This may require that the supervisor request a new SOP signature list for that staff member so that they can sign the SOP for newly assigned tasks.
- The departmental supervisor is responsible for ensuring that all outdated versions of SOPs are discarded or destroyed when the newer revisions are issued.

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

2.2.15 Note that versions of methods or SOPs which have been given to regulatory agencies or clients are generally uncontrolled in that they will not be updated except by specific arrangement. Controlled documents may be released to clients upon specific arrangement. However, the laboratory can only control the document and any updates up to the point they are provided to the client. It becomes the client's responsibility to then ensure that they are referencing the most recent versions in their own documentation.

2.3 Storage and Filing of Controlled Documents

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

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

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

2.3.4 Electronic versions of all controlled documents are also kept on file by QA. These versions are stored in an area of the laboratory network which has limited access to designated individuals. These electronic copies will be given names as closely matched as possible to their document or SOP number. Original documents and revisions will be given the extension .R00 or .R01, etc. to indicate their revision number. Should multiple files be necessary to create a given document, they will be incorporated into a subdirectory with similar naming conventions.

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

2.4 Review and Updating of Documents

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

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

2.4.2 Unless major changes to SOPs are required, SOPs should be reviewed approximately every other year. This is usually initiated by QA issuing a Document Control Form but may also be initiated by the department supervisor or other individual. QA will query the database on at least a quarterly basis in order to assure that SOP review will occur in a timely manner. QA may also issue Overdue For Review notices at any time as additional reminders. It is the responsibility of the assigned department supervisor, however, to actually see that the review/revision occurs either by themselves or by experienced staff.

2.4.3 Changes which do not require immediate update are typos or wording changes which do not inhibit the correct interpretation of the operation involved. These should, however, still be corrected during biennial review. Items which could lead to misinterpretation or incorrect performance of the methods should be corrected as soon as feasible, even if this is prior to the scheduled review date. At the time of any revision, items addressed in the "SOP Update" forms will be incorporated into the SOP. In addition, any other updates determined at the time of the review will be added. Each review will be documented on the Document Control Form (Appendix A).

Appendix A

Document Control Form

Laucks Testing Laboratories

DOCUMENT CONTROL FORM

_____ Generate new document

_____ Modify existing document

_____ Review existing document

Document No.: _____

Document Title: _____

_____

_____

Assigned to: _____ Date: _____

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

Reviewer: _____ Date: _____

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

_____

_____

_____

_____

_____

_____

QA Approval: _____ Date _____

Appendix B

SOP Database Screen

SOP # LTL-1002 | Use the [Page Up] and [Page Down] keys to browse through the Document List

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

Current Document #: LTL-1002
Revision Date: 12/14/99
CHOOSE Department: Quality Assurance
Next Revision Due: 12/14/00
Revision #: 004
Last Review Date: 12/14/99
CHOOSE Supervisor Name: Romberg, Harry
Previous Document #: LTL-0054
Document Name: Document Tracking and Control

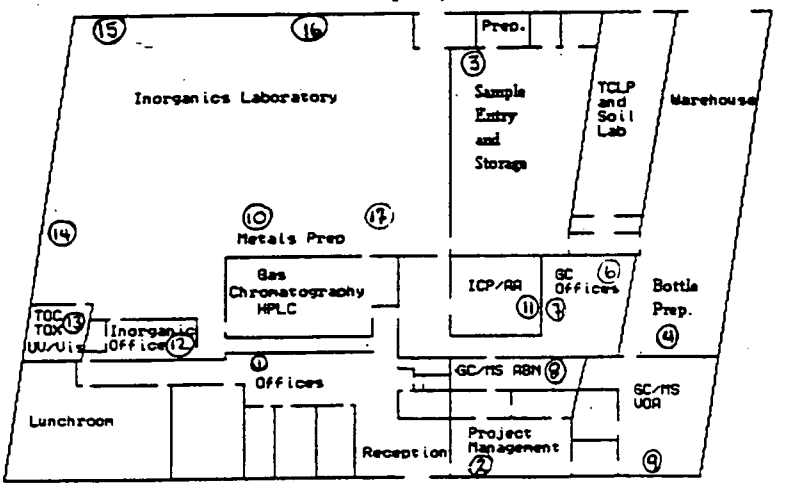
SOP Distril	Department Abbreviation
Yes	EXT
Yes	GCS
Yes	GCV
Yes	IN
Yes	MSS

Record: 1 of 14

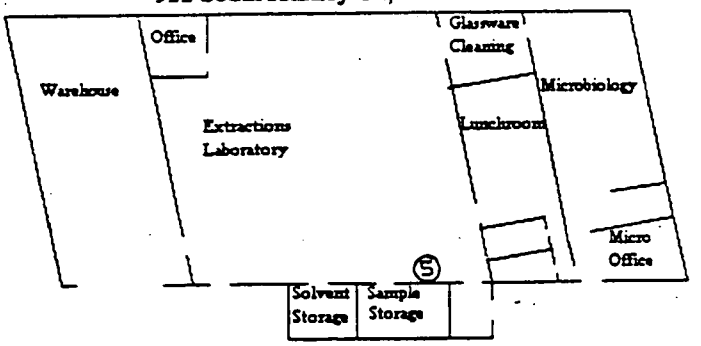
Appendix C

SOP Manual Distribution Locations

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



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



SOP Book Location

Appendix D

Document Control Stamp

Controlled Document

No. _____ Assigned to: _____

Appendix E

SOP Update Form

Laucks Testing Laboratories

SOP UPDATE FORM

Document No.: _____

Document Title: _____

_____

_____

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

_____

_____

_____

_____

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_____

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_____

Submitted by: _____ Date: _____

Approved by (QA): _____ Date: _____

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1003

Title: Chain-of-Custody and Documentation Procedures

Revision history:

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

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

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

UNCONTROLLED

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1. Introduction and Scope

1.1 Description

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

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

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

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

1.2 Definition of Terms

1.2.1 Custody - A sample is considered under custody if:

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

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

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

2. Equipment List

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

3. Safety Precautions

3.1 Safety Precautions

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

4. Operation procedures

4.1 Identification of Samples Requiring Strict Chain-Of-Custody

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

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

4.2 Initiating Internal Chain-Of-Custody

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

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

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

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

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

4.3 Maintaining Internal Chain-Of-Custody

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

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

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

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

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

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

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

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

4.5 Review of Custody Logs

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

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

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

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

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

Appendix A

Secure Storage Custody Log

Laucks Testing Laboratories, Inc.

Secure Storage Custody Log

Project:_____ **LTL Number:**_____

Number of Containers (*optional*):_____

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

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

Samples Disposed of by _____ on _____

Appendix B

Volatiles Custody Logs

LAUCKS TESTING LABORATORIES
GC/MS VOA CUSTODY LOG

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

GC Volatiles Custody Log
Laucks Testing Laboratories, Seattle, Washington

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

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1004

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

Revision history:

Number	Date
1.0	01/31/95
2.0	12/27/95
3	6/23/96
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

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1. Introduction and Scope

1.1 Description

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

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

Met Criteria Did Not Meet Criteria

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

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

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

_____ _____
Division Manager Date

Appendix C

Example Training Checklists

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

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

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

_____

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

Laucks Testing Labs
GC Volatile Analyst Training Verification Checklist

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				
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				
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-1005

Title: **Analytical Balances**

Revision history:

Number	Date
LTL-0005 R1	09/25/86
LTL-0005 R2	11/3/87
3	06/22/96
4	5/15/98
5	12/15/99

Revised by: Harry Romberg Date: 12-15-99
Harry Romberg, QA Officer

Approved by: Kathy Kreps Date: 12-15-99
Kathy Kreps, Laboratory Director

Contr[illegible] [illegible]ocument
No. 20 Assigned to: Tetra

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APPENDIX I **10**

SAMPLE PAGE FROM A BALANCE LOGBOOK 10

1. Introduction and Scope

1.1 General

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

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

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

2. Equipment List

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

3. Safety Precautions

3.1 Safety

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

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

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

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

4. Operation Procedure

4.1 Balance Setup

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

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

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

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

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

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

4.1.7 All doors to the weighing compartment should be closed.

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

4.2 Balance and Weight Calibration

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

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

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

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

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

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

4.2.2.1 The Class "S" Weights - These are the primary standards for checking the accuracy of the balance. They must be handled with care as they are calibrated and damage to the weights may result in inaccurate balance calibration. These weights must only be touched with the forceps supplied with the weights or with the clean white gloves also kept with the weights.

4.2.2.2 The class "S" weights are sent annually to a qualified weight re-certification service, currently Denver Instruments, although another qualified service is allowable. During this time the calibrations will be suspended or other Class "S" weights used (if available) until the calibrated weights return. In the case of our Seattle lab, two sets of certified weights are in use (one normally residing at the 940 building and one at the 921 building). Normally, these weights are used for calibrating balances in their respective buildings. During the annual class "S" weight checks, one set is sent to be calibrated and the entire Seattle facility uses the other weight set. When the first set is returned, the second is sent out and the entire Seattle office uses the first set until the second has been returned. This operation is coordinated annually by QA.

4.3 Responsibilities

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

- The balance is <u>clean</u> before use.
- The balance is <u>level</u> before use.
- The balance is <u>clean</u> and <u>level</u> after use.
- All weight has been removed and the balance lock lever has been returned to the proper position (for mechanical balances).

- In addition, all balances should be reset to zero when not in use.
- **Prior to use, the user should insure that the daily calibration check has been done**. If not, he or she must complete the task
- **After use, the user will insure the balance is clean and returned to the proper storage position.**
- The user will report any malfunction or failure of the daily check to the area supervisor.
- The user will mark and not use any balance which has failed calibration.

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

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

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

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

4.4 Daily Calibration Check

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

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

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

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

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

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

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

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

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

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

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

4.4.9 An example logbook page is presented in Appendix I

4.5 Weekly Calibration Check

4.5.1 The balance custodian is the person responsible for performing the weekly calibration check and reporting problems to the area supervisor or QA. The custodian may be a different person in each area and it is the responsibility of the area supervisor to ensure that a capable balance custodian has been assigned to each area for which they are responsible. It is the responsibility of the custodian to insure that the weekly check is done even if they are not present, such as for vacation, etc.

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

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

4.5.4 The balance custodian will insure the balance is clean.

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

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

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

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

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

4.5.9 An example logbook page is presented in Appendix I

4.6 Annual Calibration Check

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

5. References

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

APPENDIX I

Sample Page from a Balance Logbook

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

Done once per week

Initial + Date required

Row Heading

Daily Weights

Initial + Date required

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1006

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

Revision history:

Number	Date
1	05/01/96
2	3/20/98
3	12/21/99

Written by: Harry Romberg
Harry Romberg, QA Officer
Date: 12-21-99

Approved by: Kathy Kreps
Kathy Kreps, Laboratory Director
Date: 12-22-99

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1. Introduction and Scope

1.1 Method Description

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

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

2. Equipment List

2.1 Equipment

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

3. Safety precautions and Waste Disposal

3.1 Safety Precautions

3.1.1 During the calibration and data recording the analyst will be exposed to minimal safety hazards: boiling water, hot ovens, and mercury filled thermometers. It is incumbent on the analyst to exercise due care and caution while executing this SOP. The company will provide any protective equipment or clothing needed to assure employee safety.

3.2 Waste Disposal

3.2.1 No waste is generated in this operation. If mercury-filled thermometers are broken, however, the mercury must be collected and stored with other elemental mercury so that it may either be used in other laboratory operations or disposed.

4. Thermometer Purchasing and Identification

4.1 Purchasing

4.1.1 Laucks currently has several Streck Laboratories, ERTCO and VWR Brand thermometers for cold storage monitoring but thermometers may be purchased from any reputable supplier.

4.1.2 Thermometers used for sample or standard cold storage should be accompanied by either an actual certificate of calibration against a NIST traceable thermometer or a certificate verifying that a the thermometer was calibrated in accordance with standards traceable to the National Institute of Standards and Technology and does not vary by more than one scale division. They are immersed in a vial of ethylene glycol or equivalent solution to prevent freezing and to stabilize the temperature.

4.1.3 Thermometers used for oven temperature monitoring or for other purposes need to cover the expected range of the unit or process to be measured.

4.2 Identification

4.2.1 Thermometers are received with an individual serial number imprinted on the thermometer or may be identified in any way that distinctly distinguishes them from any other thermometer. This may involve the laboratory marking the thermometer to distinguish it from others if it does not have a distinct serial number. The use of a temperature resistant grease pen may be the most suitable for this purpose but any mechanism may be used as long as the thermometer is distinctly identified.

5. Calibration

5.1 Recalibration of the standard thermometer.

5.1.1 The NIST traceable standard thermometer is recalibrated annually by sending it back to a manufacturer who has the capability to recalibrate thermometers to NIST specifications. Currently, Laucks uses the EverReady Thermometer Company (ERTCO) for recalibration services. This vendor will re-calibrate thermometers at approximately the same points at which the original calibration was performed and will take thermometers from any vendor, as long as a copy of the original calibration certificate is available.

5.1.2 Note: Microbiology NIST traceable thermometers are recalibrated at the frequency required by the Washington State Department of Health, every 3 years.

5.1.3 At a minimum, copies of the certificates of recalibration will be kept in QA files.

5.2 Recalibration of Cold Storage and Room Temperature Thermometers

5.2.1 Refrigerator thermometers are calibrated upon receipt and annually thereafter, shortly after the return of the standard thermometer from its annual recalibration. When a thermometer has been recalibrated, a small color coded sticker is attached. The color code will correspond to a particular yearly calibration. Thus an observer can easily know his/her thermometer is currently calibrated.

5.2.2 Cold storage thermometers should not be calibrated with the standard thermometer if the standard thermometer has just been used at high temperatures (such as boiling water solutions). Thermal expansion of the thermometer at radically different temperatures may result in inaccuracies. After use at high temperatures, the standard thermometer should be allowed to stabilize at room temperature for at least 24 hours before it is used for cold storage calibration.

5.2.3 Refrigerator thermometers are placed in any functional refrigerator which is not frequently opened and has adequate space and in which the temperature is between +2°C and +6°C. Freezer thermometers are placed in a functional freezer where the temperature is between -10°C and -20°C. The temperature of the refrigerator or freezer is not especially important except that it must be accurately recorded and should be in the approximate range that refrigerators or freezers are generally be kept. Cooler thermometers are already immersed in a small vial of liquid. If a thermometer is not already in such a vial it may be placed in the same Erlenmeyer flask as the standard thermometer noted below.

5.2.4 At the same time the standard thermometer is also placed in the cold storage unit. The standard thermometer is placed in the cooler in an Erlenmeyer flask of water, ethylene glycol or other suitable liquid that will not freeze at the temperature of the unit.

5.2.5 Thermometers used for temperatures near room temperature may be calibrated in the BOD incubator using the same process.

5.2.6 The thermometers are allowed to equilibrate at least overnight (12 hours) and the temperatures read and recorded. Read the temperature of the standard thermometer first, then the individual thermometers.

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

5.2.7 Temperatures are recorded on a blank hardcopy of an Excel spreadsheet along with the cold storage unit ID and location, the thermometer ID, and the date (See appendix A). The data are later transferred to the electronic version for storage and printing. The standard thermometer and the individual thermometer readings are recorded in the log and the difference is calculated and recorded to the nearest 0.1°C.

5.2.8 The differences in temperature between the standard thermometer and the individual thermometer are calculated and recorded in the log as the "correction factor". The correction factor is calculated as the standard thermometer reading minus the individual thermometer reading so that by adding the resulting number to the individual thermometer reading will result in a "correct" temperature.

5.2.9 Correction factors are also recorded on the Cold Storage Temperature logs. An example of one of these forms is in Appendix B. The year at the top of this form changes annually without invalidating this SOP. They are located on each cold storage unit which is used for storage of environmental samples or standards.

5.2.10 When a thermometer has been recalibrated, a small color coded sticker is attached. The color code will correspond to a particular yearly calibration. A similar color coded sticker will be attached to the original hardcopy of the annual calibration log so an observer (with the log) can easily know that the thermometer is currently calibrated. Other thermometers will be marked with tape, the calibration factor noted, and initialed and dated.

5.3 Recalibration of Oven and Other Thermometers

5.3.1 Oven and other warm temperature thermometers should not be calibrated with the standard thermometer if the standard thermometer has just been used at low temperatures (such as refrigerator or freezer calibrations). Thermal expansion of the thermometer at radically different temperatures may result in inaccuracies. After use at low temperatures, the standard thermometer should be allowed to stabilize at room temperature for at least 24 hours before it is used for high temperature calibration.

5.3.2 For hot temperature calibration (generally used at temperatures too hot to touch), thermometers are calibrated in a boiling water bath. The standard and individual thermometers are inserted into a beaker of boiling water up to the immersion line. The thermometers will read a temperature slightly above 100°C if the bulbs of the thermometers are resting directly on the bottom of the beaker while the hotplate is in a heating mode. The thermometers are allowed to equilibrate for four-five minutes and the temperatures read to the nearest 1°C. Temperatures are recorded in an Excel spreadsheet along with the oven ID (if it was an oven thermometer), the thermometer ID, and the date (See appendix A).

5.3.3 The differences in temperature between the standard thermometer and the individual thermometer are calculated and recorded in the log as the "correction factor". The correction factor is calculated as the standard thermometer reading minus the individual thermometer reading so that by adding the resulting number to the individual thermometer reading will result in a "correct" temperature.

5.3.4 Correction factors may be written on the thermometer or on the unit with which that thermometer is used.

5.3.5 When a thermometer has been recalibrated, a small color coded sticker is attached. The color code will correspond to a particular yearly calibration. A similar color coded sticker will be attached to the original hardcopy of the annual calibration log so an observer (with the log) can easily know that the thermometer is currently calibrated. Other thermometers will be marked with tape, the calibration factor noted, and initialed and dated.

5.4 Recalibration of the Infrared Thermometer

5.4.1 The standard thermometer is placed in a glass Erlenmeyer flask filled with water in a cold storage unit at least overnight (12 hours).

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

5.4.3 The emissivity of the IR thermometer is set at the level determined in the previous calibration (if any). It should read the same temperature as the standard thermometer. If it doesn't, the emissivity is adjusted until the standard thermometer and the IR thermometer agree as closely as possible.

5.4.4 This emissivity setting, the calibration date and the person who performed the calibration are recorded on a label which is attached to the thermometer. Analysts subsequently using the thermometer must measure against a glass container and use the emissivity setting noted on the IR thermometer in order to get an accurate temperature measurement.

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

5.5 Daily Calibration Check of the Infrared Thermometer

5.5.1 Outside of its regular calibration against the NIST traceable thermometer, the infrared thermometer is checked daily at 4°C ± 2°C against a regular glass-column thermometer, which itself has been calibrated as previously discussed.

5.5.2 The regular thermometer is stored in the 940 walk-in (W01) in water in an Erlenmeyer flask. The infrared thermometer is checked against the side of the Erlenmeyer. The infrared thermometer should agree with the regular thermometer (corrected to the NIST thermometer) within ±2°C or the discrepancy further investigated.

5.5.3 Most often, a discrepancy may be corrected by cleaning the surface of the Erlenmeyer and re-taking the measurement or by replacing the IR thermometer's batteries. The latter or any other action must be recorded on the calibration logsheet. If discrepancies

persist but are consistent, it may just be necessary to fully recalibrate the infrared thermometer and adjust the approved emissivity setting. This should be coordinated with QA.

6. Monitoring Responsibilities

6.1 Use Of Calibration Logs For Cold Storage Monitoring

6.1.1 At the time of annual calibration of the individual thermometers, the correction factor is written in the space provided on the form by QA. This correction factor (as noted) is calculated such that adding the value results in a temperature corrected to the standard thermometer. This correction factor may be positive or negative depending upon whether the specific individual thermometer read low or high when compared to the standard thermometer.

6.1.2 It is not the intent of this SOP to discuss how individuals whose responsibility it is to monitor cold storage units are assigned. It is the responsibility of departmental supervisors to ensure that this activity is occurring in their areas.

6.1.3 The person monitoring each cold storage unit will add the correction factor to the value read on the thermometer when recording the temperature. The corrected temperature is reported to the nearest 0.1°C. As noted previously, temperatures are estimated between thermometer marks.

6.1.4 The person monitoring each cold storage unit will also check the thermometer to make sure their are no breaks in the column.

6.1.5 It is the responsibility of the person monitoring the particular unit to take corrective action as noted in this SOP and on the monitoring form or to see that corrective action is initiated by informing a supervisor. Any corrective actions (including simple adjustments of the cold storage unit thermostat) **must** be noted on the Cold Storage Temperature Log (Appendix B).

6.1.6 The calibration forms change quarterly, when new cold storage units are put on-line, or when unforeseen circumstances occur which call for a new form. The individual charged with monitoring the cold storage unit will transfer the cold storage ID, the cold storage unit location, the thermometer ID, and the correction factor to the new form. That person will also turn in the completed log to QA for permanent storage.

6.2 Monitoring Ovens And Other Devices

6.2.1 When oven thermometers are calibrated, QA will mark the oven with the thermometer ID and the correction factor. Log sheets are generally not used for ovens. It is the responsibility of any analyst using an oven to apply the correction factor when recording temperatures on data sheets.

6.2.2 In other cases where thermometers are calibrated, the correction factor will be kept with the thermometer or written directly on the thermometer (generally with a piece of tape). Again, it is the responsibility of the analyst to apply the correction factor when recording temperatures on data sheets.

7. Specification Limits and Corrective Actions

7.1 Thermometer Criteria

7.1.1 Thermometers should not vary by more than ±5°C from the standard thermometer reading, even though a correction factor is applied. This criterion does not apply to the infrared thermometer.

7.1.2 There should be no observable breaks in the column of any thermometer at any time during calibration or routine use.

7.2 Thermometer Corrective Actions

7.2.1 Thermometers with a break in the column must be immediately removed from use and either repaired or replaced.

7.2.2 Thermometers which read more than ±5°C from the standard thermometer reading must not be used. If they cannot be repaired or (if new) returned to the vendor, they should be disposed or clearly marked and only used for non-critical tasks. They should not be used for the storage or analysis of environmental samples or others where temperature is a critical factor.

7.3 Cold Storage Criteria

7.3.1 Refrigeration units should be in the temperature range of 4°C ± 2°C. All freezers must be <-10°C.

7.4 Cold Storage Corrective Actions

7.4.1 See Appendix B for an example Cold Storage Temperature Log. This log also contains the appropriate corrective actions in an abbreviated form.

7.4.1.1 If at all possible, actions should be taken **before** a unit exceeds temperature criteria so that samples or standards are not inadvertently stored outside the required limits for any significant length of time. This alleviates the far more onerous tasks of re-preparing standards or contacting clients for samples stored out-of-specifications.

7.4.2 Adjust the thermostat of the cold storage unit if necessary.

7.4.3 Defrost the cold storage unit if necessary. This may be done prior to adjusting the thermostat if there is severe icing of the unit and it is obvious that this is the cause of the temperature deviation.

7.4.4 If the above fail to correct the problem, contact the laboratory maintenance personnel, the departmental supervisor or QA to arrange for repair.

7.4.5 If it is determined that professional servicing is required this may be arranged upon direction of one of these individuals or another senior supervisor. If professional maintenance does not correct the problem, the unit may need to be replaced, again at management discretion.

7.4.6 Samples must not be stored at inappropriate temperatures. If the problem is not quickly solved, samples or standards must be transferred to another cold storage unit. If it is determined that samples were stored at inappropriate temperatures for an extended period, it may be necessary to contact clients to determine the course of action they would like us to take regarding their analyses. This should be coordinated with QA and project management. Standards which have been inappropriately stored will generally require disposal, generally at the discretion of QA and/or department managers.

7.4.7 Any corrective actions (including simple adjustments) **must** be noted on the Cold Storage Temperature Log (Appendix B).

Appendix I

Example Calibration Spreadsheet

Laucks Testing Laboratories
Thermometer Calibrations
1999

Cold Storage ID	Location	Serial No.	Cal. Date		FC-53631 NIST reading	Thermom. Reading	Correction Factor
C01	Inorganics	ERTCO 4156	05/10/1999		1.9	2.1	-0.2
C02	GC residues	none	05/10/1999		2.0	2.0	0.0
C03	extractions	B B01679	05/10/1999		2.0	1.8	0.2
C04	GC VOA	B B03987	05/10/1999		2.0	2.2	-0.2
C05	Inorganics	B02580	05/10/1999		1.9	1.5	0.4
C06	Warehouse	B B04765	05/10/1999		2.0	1.4	0.6
R02	GC Semi Stds.	B B03534	05/10/1999		2.0	1.8	0.2
R04	GC/MS whse.	B B00906	05/10/1999		2.0	1.9	0.1
R06	extractions	B B01708	05/10/1999		2.0	1.8	0.2
R07	extractions	B B04059	11/15/1999		4.0	4.0	0.0
R08	Inorganics	B03021	05/10/1999		1.9	1.8	0.1
R11	929 warehouse	B B01342	05/10/1999		2.0	2.0	0.0
W01	940 Walk-in	B B03928	05/10/1999		2.0	1.8	0.2
W02	921 Walk-in	B B01919	05/10/1999		2.0	2.1	-0.1
F03	GC semivolatiles	B B B08959	05/11/1999		-11.3	-12.0	0.7
F04	GC semivolatiles	B B B09215	05/11/1999		-11.3	-11.3	0.0
F05	GC/MS VOA	B B B06608	05/11/1999		-11.3	-12.7	1.4
F06	GC/VOA	B B B08543	05/11/1999		-11.3	-12.0	0.7
F07	GC/VOA stds.	B B B08765	05/11/1999		-11.3	-12.0	0.7
		Ertco 5236	05/11/1999		-11.3	-12.4	1.1
F08	929 warehouse	Ertco 4268	05/11/1999		-11.3	-11.3	0.0
		Ertco 5034	05/11/1999		-11.3	-12.5	1.2
Hg thermometer	Sample entry	BCR 2	05/10/1999		2.0	1.8	0.2
IR Thermometer (ITT-330)		Horiba 226099	05/10/1999		1.6	1.7	**(at E=82)**
spare		VWR 172101	05/10/1999		1.9	0.6	1.3
Oven/Water-bath/Other ID	**Location**	**Serial No.**	**Cal. Date**		**FC-53631 NIST reading**	**Thermom. Reading**	**Correction Factor**
VWR 1320		VWR 61019-204	06/22/1999	retired 6/22/99	101	103	-2.0
VWR 1320	main lab brown hood	F22641	06/22/1999		101	102	-1.0
Thelco		2	06/16/1999		101.0	101.5	-0.5
VWR 1310		3	06/16/1999		101.0	100.0	1.0
VWR 1330F	#4 Univ. Enterprises	L12-004	06/16/1999		101.2	100.8	0.4
VWR 1370F		5	06/16/1999		101.2	101.2	0.0
back waterbath (ASTM 1F)		VWR 02429	06/16/1999		101.2	211.5 F	2.7 F
front waterbath		VWR 61066-046	06/16/1999		101.2	97.2	4.0
VWR 1300 U (TOC rooom)		SPER Sci. 106	06/16/1999		101	102	-1.0

Laucks Testing Laboratories
Thermometer Calibrations
1999

Cold Storage ID	Location	Serial No.	Cal. Date	FC-53631 NIST reading	Thermom. Reading	Correction Factor
Pensky Martin (ASTM 9F)		VWR 61091-001	06/16/1999	100.8	213 F	0.4 F
VWR 1305U	TSS/TDS area	F 14547	06/23/1999	101	100	1.0
VWR 1300 U (Extractions)		F 14506	06/22/1999	100	97	3.0
Blue M (Extractions)		F 14669	06/23/1999	101	102	-1.0
front waterbath		Enviro-Safe 6	11/11/1999	99.5	102	-2.5
BOD thermometer		1	05/21/1999	2.6	2.8	-0.2
Napco Oven		SPER Sci.	06/16/1999	101.2	100.5	0.7
Digestion Area		Polyscience USA	06/16/1999	100.8	103	-2.2
VWP ASTM 9F		09107	06/16/1999	100.8	216 F	(-2.6 F)
ERDCO	Setaflash	1SF5531/85A038	06/16/1999	100.8	214 F	(-.6 F)
ERDCO	Setaflash	1SFA5531/151831	06/16/1999	100.8	213.4 F	0.0 F
spare		F22716	06/22/1999	100	100	0.0
spare		F22592	06/22/1999	101	98	3.0
spare		F22731	06/22/1999	101	97	4.0

Appendix II

Example Cold Storage Temperature Log

Cold Storage Temperature Log

Laucks Testing Laboratories, Inc.

Cold Storage ID #:_____ Thermometer ID:_____

Location:_____ Year: **2000** Correction Factor (*add this number when* ***recording*** *the thermometer reading)*:_____°C

	Month:				Month:				Month:			
Day	**Time**	**Temp.**	**Initials**	**Actions**	**Time**	**Temp.**	**Initials**	**Actions**	**Time**	**Temp.**	**Initials**	**Actions**
1												
2												
3												
4												
5												
6												
7												
8												
9												
10												
11												
12												
13												
14												
15												
16												
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18												
19												
20												
21												
22												
23												
24												
25												
26												
27												
28												
29												
30												
31												

Record Time and Temperature in the proper blocks and initial the entry each day of normal laboratory operation. Q:\TEMPLATS\LTLFORMS\REFERFRM.DOC 12/21/99

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

Corrective action includes
1) Adjust the temperature of the thermostat
2) Defrost the refrigerator or freezer
3) Contact the appropriate laboratory maintenance personnel, the departmental supervisor, and/or the QA Officer
4) One of the above may decide that professional maintenance is necessary or even that the cold storage unit must be disposed of.

Any and all actions **MUST** be recorded on this log sheet. If there is insufficient room, mark on the back of the page with the date the action occurred.

Samples **MUST NOT** be stored in units which are not maintaining the proper temperature.

Appendix III

Example Infrared Thermometer Daily Check Log

Infrared Thermometer Daily Check Log

Laucks Testing Laboratories, Inc.

Thermometer ID: Horiba 226099 Checked against standard glass thermometer: BCR 2

Location: Sample Receiving Year: **2000** Emissivity Setting: **82**

	Month:					Month:				
Day	**Time**	**Glass Thermometer Temp. (°C)**	**IR Thermometer Temp. (°C)**	**Initials**	**Actions**	**Time**	**Glass Thermometer Temp. (°C)**	**IR Thermometer Temp. (°C)**	**Initials**	**Actions**
1										
2										
3										
4										
5										
6										
7										
8										
9										
10										
11										
12										
13										
14										
15										
16										
17										
18										
19										
20										
21										
22										
23										
24										
25										
26										
27										
28										
29										
30										
31										

Record Time and Temperature in the proper blocks and initial the entry each day of normal laboratory operation. Q:\TEMPLATS\LTLFORMS\IR_CHK1.DOC 12/21/99

If IR thermometer exceeds ±2°C of the standard glass thermometer, corrective action must be taken. Corrective action includes

1) Change batteries 2) Clean erlenmeyer surface and read again 3) Contact the appropriate laboratory maintenance personnel, the departmental supervisor, and/or the QA Officer

4) If the IR thermometer is not working properly, the glass thermometer **must** be used.

Any and all actions **MUST** be recorded on this log sheet. If there is insufficient room, mark on the back of the page with the date the action occurred.

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1007

Title: Use of Instrument Records and Logbooks

Revision history:

Number	Date
1.0	9/5/91
2	5/13/98
3	6/19/00

Revised by: Harry Romberg, QA Officer Date: 6-19-2000

Approved by: Kathy Kreps, Laboratory Director Date: 06-19-00

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1. Introduction and Scope

1.1 Introduction

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

1.2 Scope

1.2.1 This SOP primarily addresses instrument run log maintenance, maintenance manuals and other logs not addressed in other SOPs. Standards log, for instance, are discussed in the standards SOP, LTL-1013. Analytical balance logs are discussed in that SOP, LTL-1005. Cold storage logs are discussed in LTL-1006. Control and monitoring of logbooks and general items pertinent to all logbooks are discussed in Laucks SOP LTL-1019.

1.3 Definition of Terms

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

2. Equipment List and Standards

2.1 Equipment

2.1.1 maintenance logbook, analytical run logbook (where appropriate) or other applicable logbook

2.1.2 pen (pencil is NOT allowed)

3. Operation procedures

3.1 All Logbooks

3.1.1 All logbooks should be numbered and controlled according to procedures outlined in Laucks SOP LTL-1019. It is the analyst's responsibility before initiating any new logbook to ensure that the logbook has been identified and given a logbook number by QA. See LTL-1019 for further detail.

3.1.2 **NOTE:** All errors in all logbooks must be altered by a single-line crossout which must also be initialed and dated. No erasures, overwriting, white-out or multiple-line crossouts (blacking out) are acceptable.

3.1.3 **NOTE:** Empty space in logbooks must be lined out (preferably with a Z for large blocks of empty space). This mark, as with error correction, should be initialed and dated.

3.2 Maintenance Manuals

3.2.1 All instruments at Laucks from GC or GC/MS systems to ICPs, AAs, spectrophotometers, ion chromatographs, etc. have instrument maintenance manuals associated with the specific instrument.

3.2.2 Maintenance manuals are bound notebooks with the specific instrument and, if appropriate where multiple similar instruments are involved, instrument names or numbers printed on the outside cover. If there are multiple books for an instrument, which may be the case for instruments which have been in service for a long time, especially if they have required extensive, ongoing maintenance, the notebooks should be clearly numbered on the cover as #1, #2, etc.

3.2.3 As a general rule, loose leaf or 3-ring bound notebooks are not acceptable. The exception to this rule is for maintaining copies of professional service call paperwork or if specific forms have been created for monitoring maintenance activities. Such paperwork must be dated in order to have a chronological record of maintenance activity that can be related to both the instrument history and sample analyses that occurred on an instrument both before and after any such activity. Note of the service should still be made in the bound notebook associated with that instrument and the identifying number on the service log noted in the maintenance manual.

3.2.4 With a few basic rules, these maintenance manuals are free-form with no specific format but MUST include any and all maintenance associated with the particular instrument.

3.2.4.1 Each entry should be INITIALED by the person making the entry.

3.2.4.2 The maintenance manual must contain the DATE any service or maintenance was performed on the instrument and exactly WHAT that operation was. This includes everything from changing a part to cleaning an instrument orifice or changing a chromatographic column or instrument tubing. It should include everything from the simplest maintenance to the most complex, including any professional service calls.

3.2.4.3 Where maintenance is routine, some books use codes for the most common service operations. These codes must be clearly defined either on the front, inside cover of the maintenance manual or on the first page. If there are multiple books, these codes must be so defined in EACH book.

3.2.5 If the maintenance was performed because of a specific problem (not just routine, ongoing maintenance) the problem should be described in at least one entry in the maintenance book as well as the work performed at any one time, and the outcome of that maintenance, that is whether or not it was successful or what occurred when the work was performed. Resolution of a malfunction should include not only that the service was successful but how it was demonstrated as having been so (i.e. the torch now lights, the oven came up to temperature, the baseline is now stable, etc.)

3.2.6 In order to aid in monitoring instrument performance changes, service or equipment changes may also be noted in instrument run logs. However, this information is supplementary. ALL maintenance must be recorded in the maintenance manual.

3.3 Instrument Run-Logs

3.3.1 Instrument run-logs come in two essentially different forms, with variations depending upon the specific instrument. In any form, a copy of the daily run log must accompany the data from each laboratory workorder for any samples associated with that sequence.

3.3.2 GC, GC/MS, HPLC, GPC and other run-logs are in bound, pre-printed, sequentially page-numbered books. They are identified by the specific instrument type and, if appropriate where multiple similar instruments are involved, instrument names or numbers printed on the outside cover. If there are multiple books for an instrument, which will be the case for instruments which have been in service for very long, the notebooks should be clearly numbered on the cover as #1, #2, etc.

3.3.2.1 Run logs must identify the method being run either at the top of the page, or if more than one method is being used for any sequence, clearly marked by the sample entry. It is recognized that it is in some cases possible to use different methods, which may only be different in the way a calibration is interpreted or validated. It may even be that two

methods are essentially identical. However, in these instances, the logsheet should clearly indicate for which method a particular sample is being analyzed.

3.3.2.2 Instrument run-logs should include places to record all relevant sample and data file IDs, performance criteria, sample type and size, additional comments pertinent to the specific analyses, and analyst initials. All appropriate information must be filled out and the page dated. Examples of current logbook forms (at the time of this writing) are located in Appendices I (GC/MS), II (GC and HPLC), and III (GPC). These forms should be considered examples and not as the only forms used by Laucks for this purpose. These forms may change with approval of the department manager and QA. Although this SOP will not then be considered invalid, new example forms should be incorporated into the next revision.

3.3.3 In addition to the appropriate header information for each analytical GC, GC/MS, HPLC, GPC or other run, all of the pertinent information should be filled out for each injection.

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

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

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

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

3.3.8 A copy of the run-log is included with each data packet associated with that run.

3.3.9 As with the bound book format, the samples, standards, calibration checks, reference materials, etc. should be identified and listed IN ORDER.

3.3.10 Information critical to identifying the analytical run (date, analyst, analysis type) must be included in the header information. If multiple analytical runs were made in one day,

they must be identified as run #1, run #2, etc. If the instrument is capable of time-stamping run data, this option should be utilized, although it need not be included in the run-log itself.

3.3.11 Where possible laboratory practice is to maintain ongoing run-logs for inorganic instrumentation. The daily run-logs are included with all data. Records which do not lend themselves to being kept in a pre-printed bound logbook may be collected in a 3-ring binder in an organized format but not unbound or loose-leaf. After sufficient logs have been collected, they should be bound with the laboratory comb binder. These logs should be given QA logbook IDs as described in Laucks SOP LTL-1019.

3.4 Other Logbooks

3.4.1 The same general principals used for the above logbooks apply to any other logbook, unless otherwise defined in a specific SOP.

3.4.1.1 Entries should be initialed and dated.

3.4.1.2 Empty space between entries should be minimized

3.4.1.3 Errors and empty spaces should be properly crossed out, initialed and dated.

3.4.1.4 Pages are preferably sequentially numbered but if this is not practical, at least dated and/or time stamped.

3.4.1.5 The logbook should identify the operation being monitored.

3.4.1.6 The pages in the logbook should contain all appropriate information needed to identify the activity and all applicable spaces should be completely filled out.

3.4.1.7 The logbook should be given a QA ID number as described in LTL-1019.

4. References

4.1.1 National Environmental Laboratory Accreditation Conference, Ch. 5 Quality Systems, 7/1/1999, Section 5.12

4.1.2 Shell for Analytical Chemistry Requirements, U.S. Army Corps of Engineers, Version 1, 11/2/98, section 4

4.1.3 Navy Installation Restoration Laboratory Quality Assurance Guide, 2/6/96, 3.1.2.10

Appendix I

GC/MS Run Logs

Lauc ing Labs, Inc.
GC/MS VOA Instrument Run Log

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

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

624 8260 524 SIM CLP-Low CLP

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

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

Laucks Testing Laboratories

GC/MS Semivolatiles Injection Logbook

IS Std ID_____

CCV ID_____

DFTPP ID_____

Page: 5699

Date:_____

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

Appendix II

GC/HPLC Run Logs

Laucks Testing Labs

GC VOA Instrument Log

Analyst : _____ Date : _____

Chromatography Ref. Page : _____ Calibration Reference : _____

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

LAUCKS TESTING LABORATORIES

INSTRUMENT LOGSHEET

Analyst : _____ Date : _____

Instrument ID : _____ Column 1: _____

Injection Volume : _____ ul Column 2: _____

Calibration Standard Reference : _____

File ID	Sample ID	DF	Comments

LAUCKS TESTING LABORATORIES

HPLC INSTRUMENT LOGSHEET

Analyst : _____ Date : _____

Instrument ID : _____ Column : _____

Injection Volume : _____ ul Solvent : _____

Calibration Standard Reference : _____

File ID	Sample ID	DF	Matrix	Comments

Appendix III

GPC Run Log

Laucks Testing Labs, Inc.
GPC Bench Sheet

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

Operating Conditions:

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

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

Comments:

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1008

Title: **QC Corrective Action**

Revision history:

Number	Date
1 (LTL-0008)	12/12/86
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4	3/3/96
5	6/22/96
6	6/16/00

Written by: Harry Romberg, Quality Assurance Officer Date: 06-19-2000

Approved by: Kathy Kreps, Laboratory Director Date: 06-19-00

Controlled Document

No. 20 Assigned to: Tetra

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1. Introduction and Scope

1.1 Purpose

1.1.1 The purpose of this SOP is to establish a system to identify, document and resolve out-of-control events.

1.2 Scope

1.2.1 An out-of-control event may be recognized by any member of Laucks. When they occur, the analyst, supervisor and Quality Assurance work jointly to solve and correct the problem. Out-of-control events are documented using an Out-of-Control-Event form or a Corrective Action form, or in a few selected instances, on a logsheet with space specifically for such actions. Corrective action resulting from an audit is also dealt with using its own Audit Response form but this action is elucidated in an SOP specific to that process.

2. Definition of Terms

2.1 This section defines terms and acronyms as they are used in this SOP.

2.1.1 **Corrective Action**: Action taken by an individual(s) to correct a problem as evidenced by either the failure of QC criteria or a more general problem which could affect performance of an analysis, the quality of service or other activity undertaken by the laboratory.

2.1.2 **Out-of-control event**: Any occurrence or condition failing to meet Laucks QC criteria or has the potential to impact data quality.

2.1.3 **QA/QC**: Quality Assurance/Quality Control

2.1.4 **QAP:** Quality Assurance Plan – The laboratory document governing overall Quality Assurance (QA) policy and guidelines. This document discusses physical assets as well as administrative aspects of the laboratory's QA program.

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

2.1.6 **RSD:** Relative Standard Deviation - The ratio of the standard deviation of a set of values to the mean of the set of values. A measure of the similarity of the values one to another.

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

2.1.8 **RPD**: Relative Percent Difference

2.1.9 **LCS**: Laboratory Control Sample

3. OUT-OF-CONTROL EVENT PROCEDURE

3.1 Identifying an Out-Of-Control Event

3.1.1 The following is a list of examples of out-of-control events. This is not a complete list of all possible out-of-control events and many of those listed may be different for different methods. Specific criteria are given in analytical SOPs or in other QA documents. If there is doubt about whether a situation is out-of-control and must be responded to, consult with Quality Assurance.

3.1.1.1 GC/MS instrument tune criteria failing to meet criteria

3.1.1.2 Initial calibration linearity, depending upon the method used for calibration, correlation coefficient <0.995 (<0.990 for some fuels analyses) or percent RSD failing to meet method specifications.

3.1.1.3 Daily and continuing calibration verification or calibration blanks outside acceptable ranges as defined in their respective SOPs.

3.1.1.4 **NOTE:** If any of the above instances (3.1.1.1-3.1.1.3) occurs, analysis is stopped. No sample analysis can occur until the event is back in control. A corrective action form does not need to be filled out for these instances if identified at the analyst level and corrected before any data are affected. Since even sample data with non-compliant QC are sometimes used to support other results even if the non-compliant data itself is not reportable, once actual samples or sample-specific QC have been analyzed, the above (3.1.1.1 – 3.1.1.3) constitute out-of-control events and must be formally addressed using the appropriate forms.

3.1.1.5 Matrix spike, surrogate spike or blank spike recoveries outside acceptable ranges.

3.1.1.6 Unacceptable RPD value for MS/MSD or duplicate samples.

3.1.1.7 Unacceptable values for LCS's and QC samples.

3.1.1.8 A method (reagent) blank containing interference or a target analyte at a concentration greater than or equal to ½ the method **reporting** limit.

3.1.1.9 **Note:** Samples which contain target analyte levels which are greater than 20 times the blank or which contain none of the offending analyte may be considered acceptable.

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

3.1.1.11 A sample depleted before all required analyses are completed.

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

3.1.1.13 Contaminated reagents and glassware.

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

3.1.1.15 Record keeping omissions, errors, and deviations from the record keeping standard operating procedures are also out-of-control situations

3.2 Responding to an Out-Of-Control Event

3.2.1 When an out-of-control event is recognized, each individual involved with the analysis in question has an interactive role and responsibility, these are as follows:

3.2.2 Analyst:

3.2.2.1 Must be able to recognize QC failure and immediately take the proper action or, if unsure of the appropriate response, notify the supervisor and work with the supervisor and Quality Assurance to solve the problem; also maintains QC charts.

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

3.2.2.3 Examine all calculations for correctness

3.2.2.4 Examine bench sheets for correctness

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

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

3.2.2.7 Re-analyze the sample

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

3.2.2.9 If these steps do not yield acceptable results, consult the supervisor.

3.2.3 Supervisor:

3.2.3.1 Must review all analytical and QC data for reasonableness, accuracy and clerical errors; also responsible for QC charts. Some of the above duties may be assigned to others, with supervisory oversight, if those others have been trained to observe the conditions which would initiate further investigation.

3.2.3.2 In an out-of-control event, the supervisor works with the analyst and Quality Assurance to solve the problem and prevents the reporting of suspect data by stopping work on the analysis in question and insuring that analyses for all suspect results are repeated, if possible, after the source of the error is determined and remedied.

3.2.3.3 If corrective actions do not yield results which meet specifications, it may be determined that sufficient action has been taken. The supervisor and QA will approve of such decisions and if it is determined that the data quality could be impacted, the supervisor will ensure that appropriate comments are reported with the data to the client.

3.2.4 Quality Assurance:

3.2.4.1 The Quality Assurance Officer or designee will work with supervisory personnel and/or analysts to solve out-of-control situations which are not routinely corrected at the bench.

3.2.4.2 In the event that an out-of-control situation occurs that is unnoticed at the bench or supervisory level, such as performance failure on a blind QC sample, Quality Assurance will notify the supervisor, help identify and solve the problem where

applicable, insure the work is stopped on the analysis and no suspect data is reported.

3.2.4.3 Finally the Quality Assurance Officer or designee must review and approve all corrective action reports which cannot be resolved. If corrective actions do not yield results which meet specifications, it may be determined that sufficient action has been taken. The supervisor and QA will approve of such decisions.

3.2.4.4 If it is determined that the data quality could be impacted, the supervisor will ensure that appropriate comments are reported with the data to the client and QA will review said comments.

3.2.5 Project Manager:

3.2.5.1 The Project Manager is responsible for notifying the client of out-of-control events, such as missed holding times, raised reporting limits, matrix interferences, etc. which cannot be resolved without potential impact on either the data quality, the agreed upon or routinely reported results, or the timely and expected delivery date. It is not necessary to contact the client for events which are correctable and do not impact the final data quality, holding times or turn-around unless specifically requested by the client.

3.3 Corrective Actions

3.3.1 Appropriate corrective action depends on the type of analysis, the extent of the discrepancy, and whether the event is determinant or not. The corrective action to be taken for analytical QC failures is usually described in the specific analytical method but may also be determined by either the supervisor, Quality Assurance Officer, or by both in conference, if necessary.

3.3.1.1 Some items may not necessitate direct intervention of QA where standard practices are in place for some events, where the SOP or project or program QAP itself dictates the corrective action and where the action taken is the most conservative response practical. These types of events may be considered to have automatic QA approval and may not even require the completion of any related out-of-control event forms.

3.3.2 A corrective action can be as extensive as replacing a complete lot of contaminated extraction solvent, re-extracting and re-analyzing a complete batch of samples, due to reagent blank contamination; or as simple as recalculating a series of results because a

wrong dilution factor was applied. Again, the appropriate corrective action must be determined on a case by case basis.

3.3.3 Data cannot be released until the system is in control or the QC failure can be attributed to a cause other than method performance. In the case where the out-of-control event is due to matrix problems in the sample and the system remained out of control, the data is flagged, narrative comments usually made in the report, and supporting documentation is released to the client.

3.3.4 Corrective actions are considered adequate when the problem has been resolved and data can be reported or other actions taken from an in-control condition. Alternatively, it may be determined that the action taken was, as a minimum, all that was required by the method or that no further action was reasonable or possible that would improve the data. In these cases, the final decision must be approved by the supervisor and QA.

3.4 Documenting an Out-Of-Control Event

3.4.1 This is accomplished by completing one of the following

- A Corrective Action (CA) Form (See Appendix 1)
- A QC_DB Report Form (for Inorganics analytical QC only, see Appendix 2)
- An Out-Of-Control Event (OOCE) Form (lab use only, see Appendix 3)
- A Sample Receipt Form (for sample receipt events, see Appendix 4)
- An Audit Finding Report Form (QA use only, not shown here, see audit SOP)
- or logged onto a form which itself includes corrective actions (example, Cold Storage Logsheet, see Appendix 5).

3.4.2 CA forms are general and are for documenting corrective action taken to correct problems not associated with a particular analytical event. Such events are usually more general, such as PE sample failure, questioning a general practice even if it did not necessarily precipitate an error because it could lead to problems (i.e. failing to properly document standards or misuse of a run log, etc.)

3.4.3 Out-Of-Control Event (OOCE) Forms are filled out by **technical** laboratory staff only and are designed for documenting analytical QC failures and associated corrective actions. Where other forms, such as the Inorganics QC_DB Report Form, are used to document that the QC parameters were checked, any failures of QC and the decision to perform corrective action or continue data processing must be documented on the OOCE form. The checklist may then be attached to the OOCE form for final data submission.

Note: It is not necessary for analytical staff to document actions which were taken prior to processing samples or which do not in any way affect reported data.

3.4.4 Audit Finding Reports are responded to by the assigned individual and signed off by QA or a designated individual (see the audit SOP).

3.4.5 All OOCE and Corrective Action Forms shall be filled in completely by the person observing the event. Actions taken may be filled in by either the initiating person or the person actually performing the corrective action. The descriptions of the event and any corrective actions taken should be detailed and specific. The OOCE form provides check boxes for most analytical events.

Note: Holding time violations due to laboratory error are annotated on the OOCE form. Holding time violations occurring due to receipt of samples beyond the criteria are documented on the sample receipt form only.

3.4.6 If the corrective action taken and annotated on the OOCE Form resolves the problem and allows data to be reported which is in control, the action is complete and only needs to be signed by the individual taking action and the individual initiating the action.

3.4.7 If the corrective action taken and annotated on the OOCE Form does not resolve the event and it is determined that no further action can or will be taken, the form must be signed by the analyst, supervisor, and QA.

3.4.8 Originals of all OOCE forms must be turned into QA. Copies must be included in each SDG or workorder in validatable packages and in the first workorder in the "samples affected" column for non-validatable data packages.

3.4.9 Any corrective actions taken which could either impact data directly, help to explain analytical decisions that were made in order to resolve analytical discrepancies, or which would help in the interpretation of the final data package must also be narrated in the final report. OOCE forms must be turned in with the data and the supervisor creating the narrative comment for that area will comment on any decisions resulting from failed QC which could impact data validity or interpretation.

Appendix 1

Corrective Action Form

Laucks Testing Laboratories
Corrective Action Report

1) Problem Description:

Response tasked to: _____ on _____

By: _____ Response Requested By _____

2) Cause:

3) Action Taken:

Completed by _____ on _____

☐ Corrective actions will be reviewed 30 days after completion to verify problem has been corrected.

☐ No further action necessary

Reviewed by: _____ on _____

1) Person initiating corrective action fill out Part 1 and may fill out Part 2 if they are aware of the cause
2) Original goes to person tasked with a response; one copy goes to QA Officer and another kept by person initiating corrective action
3) Person tasked completes response in Part 2 (if not previously completed) and Part 3, signs response, and returns original to person initiating action
4) Person initiating action determines if action corrects the problem and signs "Reviewed by." If action was insufficient, return to the person charged with responding without signing.
5) Completed original goes to QA Officer

Appendix 2

QC_DB Report Form

Laucks Testing Laboratories

QC_DB Report Form

Analyst_____

Checker_____

Test Code_____

		QC Exceeds Control Limit √ if yes	Corrective Action Approved By ____
PBlk	B_____00_____	☐	
MS/MSD	K_____00_____	☐	
SRM	R_____00_____	☐	
Blk Spk	S_____00_____	☐	
MS/Dup	M_____00_____	☐	
Duplicate	D_____00_____	☐	
BS/BSD	L_____00_____	☐	

This report validates the following work orders

Appendix 3

Out-Of-Control Event Form

OUT OF CON L EVENT FORM

No: _____

Date Recognized: _____
Date Occurred: _____
Method: _____
Initiated By: _____
Analyst: _____

- ☐ GC/MS VOA
- ☐ GC/MS ABN
- ☐ GC VOA
- ☐ GC non-VOA
- ☐ HPLC
- ☐ Metals
- ☐ Wet Chemistry
- ☐ Extractions
- ☐ Data Management

Samples Affected (Workorder & Sample Numbers.)

Type of Event: (*check all that apply*)

___ Holding time missed (*describe below*)
___ Blank ≥ MDL___ RL___ CRQ/DL___
___ Spike Recoveries do not meet criteria
___ Duplicate RPDs do not meet criteria
___ MS/MSD Results do not meet criteria ___ %Rec ___ RPD
___ BS/BSD Results do not meet criteria ___ %Rec ___ RPD
___ Analytical Spike recoveries do not meet criteria
___ Standard Additions do not meet criteria
___ LCS or Blank Spike Recoveries do not meet criteria
___ Surrogate Recoveries do not meet criteria
___ Calibration Corr. Coefficient does not meet criteria
___ Calibration Verification does not meet criteria ___ Init ___ Cont.
___ Recovery ___ Retention time ___ %D
___ Tuning fails criteria
___ ISTD fails criteria
___ Calculation/Transcription error
___ Other (explain)

_____
_____
_____
_____

Corrective Action: (*check all that apply*)

___ Repeat Calibration
___ Made new standards
___ Reanalyzed, Date: _____
___ Sample(s) Redigested/Reextracted Date: _____
___ Results Recalculated
___ Cleaned System
___ Ran Standard Additions
___ Notified Client _____
___ Other (Please explain)

_____
_____
_____
_____

Check One:

___ Original Results Reported
___ Rerun Results Reported

Notified:

___ QA
___ Client Services

Action taken By: _____ Date: _____ Reviewed by Initiator: _____ Date: _____

___ Out of Control Event Corrected By: _____

_____
_____
_____

___ Corrective Actions Not Successful (signatures required) **DATA MUST BE FLAGGED AND/OR NARRATED**

Analyst: _____ Date: _____
Supervisor: _____ Date: _____
QA Department: _____ Date: _____

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

Appendix 4

Example Sample Receipt Forms

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

Initial once samples are checked in_____

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

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

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

- ° (1) Not enough sample sent for proper analysis. #s affected:_____
- ☐ (2) Sample Bottle received broken and/or cap not intact._____
- ☐ (3) Custody seal: Absent____ Present/Intact_____ Present/Broken _____
- ☐ (4) Any temperature out of compliance:_____
- ☐ (5) Sample received outside of holding time._____
- ☐ (6) Sample not properly preserved. pH = ___. Wrong preservative used._____
- ☐ (7) Illegible sample numbers or label missing from bottles._____
- ☐ (8) Identification on bottle same as identification on paperwork: yes:_____no:_____
- ☐ (9) Incomplete instructions received with sample(s), i.e.,
- ☐ no Request for Analysis, no Chain-of-Custody._____
- ☐ (10) Samples received in improper container._____
- ☐ (11) Samples held in field before receipt by Lab. Days (specify)_____
- ☐ (12) Air Bubble(s) in ___of ____ samples for volatiles analysis._____
- ☐ (13) Other _____

CORRECTIVE ACTION: (Check applicable item(s))

		Correction action taken by: Inititals	Date
☐	(1) Client informed verbally (Client Services).	_____	
☐	(2) Client informed by memo/letter/fax (Client Services).	_____	_____
☐	(3) Sample processed "as received" (Sample Entry).	_____	_____
☐	(4) Re-sampling requested of client (Client Services).	_____	_____
☐	(5) Samples placed "on hold" until further notice (Sample Entry/Client Services).	_____	_____
☐	(6) NOTE IN NARRATIVE. See temperature/pH login sheet. (Sample Entry).	_____	_____
☐	(7) Other (Specify)_____	_____	_____

* When complete (within 24 hours of nonconformance) forward to QA. Original to be forwarded to initiator to be included in transmittal file.

Comments:

_____

_____

_____

_____

_____

Laucks Testing Laboratories, Inc.
SAMPLE RECEIPT LOG (2) In-house

P:\PM\Holly\docs\fax\pjmemo.doc12/22/94

DATE RECEIVED:_____ WORKORDER #:_____
CLIENT NAME:_____
RECEIVED BY:_____

Non-Conformance:

☐ Other

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

CORRECTIVE ACTION: (Check applicable item(s))

Correction action taken by:

		Inititals	Date
☐	(1) Client informed verbally (Client Services).	_____	
☐	(2) Client informed by memo/letter/fax (Client Services).	_____	_____
☐	(3) Sample processed "as received" (Sample Entry).	_____	_____
☐	(4) Re-sampling requested of client (Client Services).	_____	_____
☐	(5) Samples placed "on hold" until further notice (Sample Entry/Client Services).	_____	_____

* When complete (within 24 hours of nonconformance) forward to QA. Original to be forwarded to initiator to be included in transmittal file.

Comments:

_____

_____

_____

_____

_____

Appendix 5

Example Logsheet (Cold Storage)

Cold Storage Temperature Log

Laucks Testing Laboratories, Inc.

Cold Storage ID #:_____ Thermometer ID:_____

Location:_____ Year: **2000** Correction Factor (*add this number when* ***recording*** *the thermometer reading)*:_____ °C

Day	Month: Time	Temp.	Initials	Actions	Month: Time	Temp.	Initials	Actions	Month: Time	Temp.	Initials	Actions
1												
2												
3												
4												
5												
6												
7												
8												
9												
10												
11												
12												
13												
14												
15												
16												
17												
18												
19												
20												
21												
22												
23												
24												
25												
26												
27												
28												
29												
30												
31												

Record Time and Temperature in the proper blocks and initial the entry each day of normal laboratory operation. Q:\TEMPLATS\LTLFORMS\REFERFRM.DOC 12/21/99

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

Corrective action includes
1) Adjust the temperature of the thermostat
2) Defrost the refrigerator or freezer
3) Contact the appropriate laboratory maintenance personnel, the departmental supervisor, and/or the QA Officer
4) One of the above may decide that professional maintenance is necessary or even that the cold storage unit must be disposed of.

Any and [illegible]ns **MUST** be recorded on this log sheet. If there is insufficient room, mark on the bac[illegible] page with the date the action occurred.

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

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1009

Title: **Blind Spike / PE Sample Evaluation Program**

Revision history:

Number	Date
LTL-0048 Rev1.1	5/18/92
2	6/21/96
3	6/19/00

Written by: Harry Romberg
Harry Romberg, QA Officer
Date: 6-19-2000

Approved by: Kathy Kreps
Kathy Kreps, Laboratory Director
Date: 06-19-00

Controlled Document
No. 20 Assigned to: Tetra

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1. Introduction and Scope

1.1 Description

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

1.1.2 Materials may be from a multitude of sources. The analyst will most often be aware that the sample is a blind spike but in no case should the analyst know the "true" value of the submitted sample. On occasion, at the discretion of QA, a double blind sample may be submitted (one which the analyst does not know is an evaluation sample).

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

1.2 Definition of Terms

1.2.1 **Blind Spike** - A proficiency sample which may or may not be known as such by the analyst but which contains a target analyte with a value which is not known.

1.2.2 **Double-Blind Spike** - A proficiency sample which is submitted to the analyst in such a way that it is thought to be a routine sample and which contains an unknown amount of target analyte.

2. Equipment List and Standards

2.1 Equipment

2.1.1 Pipets, flasks, containers etc. necessary to prepare spikes for submission.

2.2 Reagents

2.2.1 Deionized water, methylene chloride and other solvents or preservatives that may be required to prepare spikes. Some samples may be prepared by outside sources and only need to be submitted to the analyst.

3. Safety precautions and Waste Disposal

3.1 Safety Precautions

3.1.1 All standards, samples and sample solutions should be handled as if they are hazardous substances. During the preparation of blind spikes, the analyst will be exposed to a variety of reagent chemicals and solvents. In addition, preservatives contained in both reference materials and in sample bottles may pose health hazards. The health effects of these various chemicals may be ascertained by reading the appropriate material safety data sheets (MSDS). It is incumbent on the analyst to exercise due care and caution while executing this SOP. The company will provide any protective equipment or clothing needed to assure employee safety.

3.1.2 Many solvents also pose a fire hazard and should be treated with proper precaution.

3.2 Waste Disposal

3.2.1 Waste solvents are disposed in the appropriate waste solvent container.

3.2.2 No more blind spike material is used than is necessary for submittal of the sample so that it will not present a disposal hazard.

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

4. Materials

4.1 Sources

4.1.1 Materials may be WS, WP or other materials from an external performance evaluation. Although these are not generated directly by the laboratory, they are blind samples in that the expected values and in many cases the constituents themselves are not known to the analyst beforehand.

4.1.2 Standard materials may be purchased from a vendor, such as Environmental Resource Associates (ERA), Analytical Products Group (APG), SPEX, Restek, Supelco or any other reputable vendor.

4.1.3 Materials may be purchased either as Performance Evaluation samples (values unknown to the laboratory), reference materials (values known to the laboratory), or as standard materials (values known to the laboratory). They may also be made up by supervisory or QA staff from materials of known content. In any instance, the value of the components

will be unknown to the analyst performing the analysis until completion of the evaluation.

4.2 Storage

4.2.1 Materials are stored as recommended by the manufacturer, most often at a temperature of 4°C ± 2°C. Metals will generally be stored in dilute nitric acid and need not be refrigerated.

5. Operation procedures

5.1 Requirements and Scheduling

5.1.1 These requirements may be program and/or method-specific. Laucks specific training requirements and documentation are discussed in other SOPs and in the QA Plan. This SOP is intended primarily to document the practices and evaluation of results and not to dictate the specific analyst requirements.

5.1.2 Initially (as part of being considered able to independently perform an analysis), an analyst may be required to analyze a single blind Performance Evaluation (PE) sample. The analyst must process the samples independently, without direction or assistance in order to be considered proficient.

5.1.3 On an ongoing basis, at least annually, an analyst may also be required to demonstrate continuing performance by analyzing a single blind PE sample.

5.1.4 PE results may also be used as a supplement to a method verification process in order to verify the laboratory's ability to perform a method.

5.1.5 These PE samples may be from a performance evaluation study, such as a Water Pollution (WP) or Water Supply (WS) study, an independent vendor PE, such as Environmental Resource Associates (ERA) or Analytical Products Group (APG), or it may be prepared by an area supervisor from a known material. Blind PE samples will almost always be prepared as aqueous solutions except in limited circumstances, such as fuel hydrocarbons, where soil samples are periodically analyzed. Other ERA, APG or independent sources of materials will be used where components are not present in WP, WS, DMRQA or other "official" PE samples. Acceptable results from programmatic samples, such as those for AIMTech, Army Corps of Engineers, or NFESC may be used to qualify analysts or to otherwise demonstrate performance, even though in some instances an actual value may not be provided by the agency.

5.1.6 WP and WS program samples are currently analyzed semiannually and DMRQA samples approximately annually. Supplementary PE samples for analytes not present in these samples (such as fuels or GC/MS semivolatiles) are generally obtained from the same source (APG, ERA or a similar vendor). Other external PE samples from programs such as NFESC, AIMTech, or the Army Corps of Engineers may be analyzed at the discretion of those programs but be used for evaluation. The precise schedule for submittal of all but programmatic samples is at the discretion of QA in order to meet laboratory needs to qualify analysts or methods or to meet other requirements. At the time of the latest revision of this SOP, both state and federal agencies are still determining the implication to their programs of WP, WS & DMRQA sample privatization. In addition, the laboratory is still evaluating the costs associated with purchasing these samples commercially. Once the requirements, costs and scheduling become more clear the result may be that there will be an overall reduction in the total number of PE samples the laboratory purchases in order to fulfill program requirements within a reasonable budget and the schedules may be modified at the discretion of QA.

5.1.7 One set of PE samples may be used to qualify several analytical staff. For instance, one person may extract a sample and be so qualified. Several analysts may process the extract independently and also be qualified. If multiple analysts do process the extract, however, there must be no collaboration between analysts until the results have been received by QA.

5.1.8 In any instance, the values of the components must not be divulged to the analyst(s) prior to analysis. Furthermore, if a PE sample contains one or more components from a multi-component analysis (such as a semivolatiles or pesticide mixture), the analytes themselves must not be divulged.

5.1.9 Blind spikes should be analyzed in at least duplicate, and frequently quadruplicate if used for analyst qualification, so that reproducibility can be determined as well as recovery. All results should be reported for each determination where the analysis was otherwise in control. Evaluation of replicates is a laboratory option and is rarely required of any external performance evaluation program.

5.1.10 Blind spikes are typically determined for the following analyses (in water excepts as noted):

- ICP metals
- ICP/MS metals
- Graphite furnace metals (optional)
- Mercury

- GC Volatiles
- Gas/BTEX water & soil
- Diesel water & soil
- Petroleum Hydrocarbons (418.1) water & soil
- Pesticides
- GC/MS Volatiles
- GC/MS Semivolatiles
- PNAs
- Cyanide
- Total Organic Halogens
- Total Organic Carbon
- Phenolics
- Ion Chromatography (F, Cl, NO_3, SO_4)
- NO_3/NO_2 Automated Cd reduction
- others at the discretion of QA

5.1.11 Where other method references are very similar to those above, the same PE analysis may be considered adequate documentation for both methods. Other blind PE studies may be conducted at the discretion of QA.

5.1.12 Samples will be given a laboratory ID number and test code when they are submitted to the laboratory and should be tracked in the same manner as a routine sample. Results will be compared against vendor-supplied, method-specific, or laboratory-derived limits as noted in the Evaluation and Reporting section.

6. Evaluation and Reporting

6.1 Data Package Organization

6.1.1 Paperwork must be completed as it would for routine samples, documenting preparation, calibration, and analysis and quality control. In addition, a summary page must be completed with the results of the sample and any replicate analysis. The summary page must contain the following elements:

- Analyst
- Date of analysis
- Preparation Technician (where appropriate)
- Date Prepared
- Analysis (Method*)

- Preparation (Method*)
- Components obtained from the analysis
- Results obtained from the analysis
- Replicates (where applicable) and associated RPDs

* At the discretion of QA, analysis and preparation methods may be considered sufficiently similar to qualify for more than one reference technique.

6.2 Evaluation

6.2.1 The data will be evaluated by QA with possible assistance from other supervisory staff. Data must meet the limits supplied by the vendor, if purchased or supplied as part of a PE program. If limits are not given by the vendor, method specific limits may be adopted or the laboratory may choose to accept recoveries based on internal QC limits.

6.2.1.1 All relevant components must be identified by the analyst, although in a few limited cases, similar components react in much the same fashion (i.e. similar retention times or patterns). In these instances, at the discretion of QA, the analyst may be allowed to re-evaluate the analysis.

6.2.1.2 If the analysis is a multi-component mixture, the results may be considered acceptable if 90% of the target analytes are quantified correctly as long as all are at least identified.

6.2.1.3 Replicates will most often be evaluated where recovery exceptions occur or where it is determined by QA or the area supervisor that this reproducibility is a critical part of the analyst's evaluation. They will also be evaluated if it is so specified in the reference method. In these instances, the acceptability criteria are generally either the laboratory-derived RPD(s) or the reference method-specified criteria.

6.2.1.4 At the discretion of QA, the data may also be evaluated for completeness and documentation.

6.3 Remedial Actions

6.3.1 If the limits for the analyzed material have been exceeded, that performance criterion will be considered to have not been met. In such case, the data will first be re-evaluated by the analyst. If sufficient extract/digestate remains, this may include re-analysis.

6.3.2 If, after re-evaluation, the performance criterion still has not been met, the results from the entire analysis will be evaluated and if sufficient criteria have not been met, the analyst may be required to analyze another blind PE sample.

6.3.2.1 In some cases, the quality of the vendor-supplied material may be in question. In this instance or in the case where no more of a specific material is available in a timely fashion, a second source of performance evaluation material may be used.

6.3.3 Continued failure may result in either or both examining the analysis/preparation method for discrepancies or it may require re-training of the analyst if it is determined that the method and instrumentation is functioning properly. In either case, action must be initiated immediately to insure that accurate results are being produced for actual laboratory samples.

6.3.4 In the extreme case, it may be determined after consultation with supervisory staff and laboratory management (including QA), that no analyses can be performed using that method or that analyst until there is demonstration of adequate performance.

7. Record Keeping

7.1 Analyst and Method

7.1.1 Records for all evaluations will be maintained by QA. Analyst evaluation will be maintained in the analyst's training file. Method evaluations will be kept separately but may mirror the analyst's evaluation.

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1011

Title: **Procedures for the Determination and Reporting of Detection Limits, Reporting Limits, Precision and Accuracy Studies, and Control Limits**

Revision history:

Number	Date
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2	1/08/96
1.1	6/15/90

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

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

UNCONTROLLED

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1. Introduction and Scope

1.1 Overview

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

1.1.2 In general, detection limits are the minimum amount of a target analyte that can be measured and determined to be greater than zero with a known degree of confidence. For purposes of this SOP, the known degree of confidence for MDLs will be defined as the 99% level. IDLs are based strictly on instrument response and MDLs on a sample processed through the entire preparation process. This SOP is based on information provided in 40 CFR Part 136, Appendix B, *Definition and Procedure for the Determination of the Method Detection Limit, Revision 1.11* and in other sources such as the EPA Contract Laboratory Program (CLP) Inorganic Statement of Work (SOW) and SW846.

1.1.3 Criteria for Precision and Accuracy (P&A) Studies are generally defined in the specific published method, particularly those in SW 846. Where criteria are not so defined, Laucks has chosen to either use the criteria from similar methods or to set in-house criteria based on the judgment of senior management and QA. Where two methods are the same in technical detail and one does not provide P&A criteria, performance under the guidance of the method with specifications may be used to satisfy the performance criteria of both.

1.1.4 Control limits are determined initially for an analysis, generally using limits supplied in the method or defined by the program (such as CLP). After sufficient points have been accumulated the laboratory performs a statistical analysis of the data and computes the control limits which are based on 3x the standard deviation of recoveries (for accuracy limits) or relative percent differences (for precision limits). In some instances, warning limits may also be established using 2x the appropriate standard deviation.

1.1.5 This SOP is designed for applicability to a wide variety of sample types ranging from reagent water to solids containing the analyte. The MDL may vary as a function of sample type. Laucks rarely determines MDLs on any matrix other than soil or water. Other MDLs may be estimated based on these studies.

1.1.6 This SOP requires that a specific, detailed analytical method exist. When determining MDLs and P&As following this SOP, it is imperative that all sample processing steps included in the analytical method be included.

1.1.7 Where a specific method has requirements exceeding the requirements of this SOP, that method will take precedence. Where a reference method has stated detection limits, these are generally taken to be MDLs. This SOP is to be followed to validate a new method or to validate a change in a current method.

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

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

1.1.10 This method is restricted to use by, or under the supervision of analysts experienced in the technique described. Each analyst performing this method must have demonstrated the ability to perform the described analysis except in the case of P&A studies which are used to demonstrate the competency of the analyst.

1.2 Method Description

1.2.1 Detection Limits

1.2.1.1 For any metals method, the Instrument Detection Limit (IDL) must first be determined. The IDL may also be determined strictly for informational purposes for other methods but is not required. The IDL allows the analyst to assess the precision of the measurement system and to estimate the target concentration for the MDL study. IDLs are generally determined by analyzing 7 low-level standard replicates on 3 non-consecutive days and averaging the sample standard deviations from each of the three days.

1.2.1.2 In order to determine MDLs, a minimum of seven replicate measurements are made of a prepared sample matrix which contains approximately 1 to 5 times the estimated detection limit. A Student's t determination is made for the number of data points available, usually 7 (6 degrees of freedom), and the resulting standard deviation multiplied by that value to determine the MDL. All MDL data are entered into the laboratory MDL database.

Note: The CFR states that the recommended concentration levels used to determine the MDL be one to five times the MDL. It later implies that a level of up to 10 times the MDL is acceptable. Laucks considers up to 10 times the MDL to be an appropriate concentration although limited exceptions to this rule may be granted as long as the deviations are not great and they are approved by QA.

1.2.1.3 Reporting Limits (RLs) are set by the laboratory as limits that can be reliably reported on a consistent basis with a reasonable degree of confidence that the reported level is accurate. These limits may be set at the Practical Quantitation Limit (PQL) initially by using a multiplier times the MDL. The multiplier is often but not always defined in the method. After initial setting of the RL, it is rarely changed unless significant changes in the MDL occur which make it necessary to raise or lower the RL.

1.2.2 Precision and Accuracy (P&A) Studies are studies performed in order to demonstrate the laboratory's ability to perform a method and are also used to demonstrate analyst competency to perform the method. They generally involve the analysis of 4 replicates spiked at concentrations defined in the method. Where no method guidance is provided, the replicates should be prepared at concentrations of 10 to 50 times the MDL for each analyte. Adequate performance is most often defined in the reference method, although if the method performance has been demonstrated, analyst competency may be demonstrated in comparison to laboratory limits.

1.2.3 Control limits may be specified in a reference method or may be statistically determined by the laboratory from existing data. In general, laboratory determined limits for control samples must not exceed method specified limits. If laboratory determined limits do exceed method-specified limits, the entire system must be evaluated to improve method performance. In most instances, it is unacceptable for routine performance to exceed method-specified performance even if the laboratory is using method-specified control limits. This is because the laboratory cannot demonstrate adequate performance for all samples on a routine basis.

1.2.4 It is not uncommon for clients to specify reporting or control limits in their project quality assurance plans. As long as they are achievable (i.e. the requested RL is not lower than the laboratory MDL), Laucks will generally comply with the client's request for that particular project.

1.3 Definition of Terms

1.3.1 **Accuracy** - The degree of agreement of a measurement (of an average of measurements of the same thing), X, with an accepted reference or "true" value, T, usually expressed as

the difference between the two values, X-T, or the difference as a percentage of the reference or true value, 100*(X-T)/T, and sometimes expressed as a ratio, X/T. Accuracy is a measure of the bias in the system. Accuracy shall be calculated as follows:

$$\%R = \frac{C_s - C_u}{S} * 100$$

Where:

Cs = Concentration of spiked sample
Cu = Concentration of unspiked sample
S = Expected concentration of spike in sample
%R = Percent recovery

1.3.2 **Control Limits** - Control limits may be specified in a reference Method (either as mandatory or guidance limits), or may be developed by the laboratory using internal performance data. Control limits represent acceptance criteria for determining whether an analytical system is in control (functioning within acceptable guidelines).

1.3.3 **Control Sample** - A QC sample introduced into the analytical process to allow evaluation of the measurement system. In general, it is best to use samples of a matrix similar to the samples being analyzed, where such are available. The control sample, however, will generally be free from interferences other than those inherent to the matrix itself.

1.3.4 **Degrees of Freedom** - The number of independent estimates that could be obtained from a specific set of data. In general, for a simple set of *n* independent values,

$$df = n-1$$

1.3.5 **IDL** - Instrument detection limit - The lowest concentration of a target analyte that can be measured and known to be greater than the instrumental background with a known degree of confidence. It may be used as a starting point for selecting MDL study spiking levels.

1.3.6 **MDL** - Method detection limit - The minimum concentration of a substance that can be measured and reported with a known degree of confidence (99% for our purposes) that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.

1.3.7 **Mean** - The arithmetic sum of a set of observations divided by the number of observations.

$$\frac{\sum X_i}{n}$$

Where:

X_i = sample value for replicate *i*
n is the number of replicates

1.3.8 **P & A** - Precision and Accuracy - This often refers to a study conducted to validate a method or an analyst conducting a particular method.

1.3.9 **PQL** - Practical Quantitation Limit - The limit at which it is determined that the constituent can not only be detected but be accurately quantified. This limit is usually 2 to 10 times the MDL but may be even larger depending upon the constituent and the matrix. Factors are often taken from the published method but may be set by the laboratory if published factors do not exist. These limits may also be used as the routine reporting limit (RL), unless otherwise contractually defined.

1.3.10 **Precision** - A measure of mutual agreement between individual measurements of the same property, usually under prescribed similar conditions. Precision is best expressed in terms of the standard deviation. Various measures of precision exist depending upon the "prescribed similar conditions".

1.3.11 **Reporting Limit (RL)** - A value greater than or equal to the MDL or the IDL which may be based on QA decision, the published method specifications, or project-specific requirements.

1.3.12 **Standard deviation** - A statistical measure of the variability of a set of sample observations. For the purposes of this SOP, the sample standard deviation is used. This is calculated using the formula:

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

Where:

s = the standard deviation estimated with n-1 degrees of freedom.
X_i = sample value for replicate *i*
$\overline{X}$ = mean of all of the replicates
n = the number of replicates

2. Equipment List and Standards

2.1 Equipment, Reagents and Standards

2.1.1 As appropriate for the given analysis.

2.1.2 Personal Computer with access to a spreadsheet program such as *Microsoft Excel* and the laboratory MDL database.

3. Safety precautions and Waste Disposal

3.1 Safety Precautions

3.1.1 Refer to the specific analytical SOP for appropriate safety precautions.

3.1.2 Waste Disposal

Refer to the specific analytical SOP for appropriate waste disposal practices. Waste segregation and disposal from the point of collection is further covered in the Laucks SOP on Waste Segregation and Disposal.

4. Calibration and Quality Control

4.1.1 Calibration is as appropriate to the specific method. No matrix spiking or other routine QA is required.

5. Responsibilities

5.1 Analyst

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

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

5.1.3 A metals analyst is responsible for assuring that a quarterly IDL study is produced on each instrument.

5.1.4 Each analyst is responsible for labeling MDL and P&A studies appropriately.

5.1.5 Each analyst is responsible for turning in a legible MDL, IDL, and P&A study to their supervisor for review and approval prior to final submittal to QA.

5.1.6 All of the analyst activities should be coordinated through the area supervisor.

5.2 Supervisor or Senior Analyst

5.2.1 Each area supervisor or senior analyst is responsible for coordinating the effective completion of the required studies. This may include but not necessarily be limited to helping determine appropriate concentration levels, coordinating the completion of the study within the timeline required by the method and/or the QA department, and scheduling the study around the analytical workload.

5.2.2 It is the responsibility of the area supervisor or senior analyst to insure that the analyst is performing the study within the guidelines of the method and to perform a review of the final data prior to submission to QA. This review should include determination that appropriate spiking levels were used, that the data was properly computed and transcribed, and that any problems or concerns encountered during the study are documented. Part of this review must include the comparison of the data to method specific criteria. In other words, P&A data must be compared to established method criteria and MDLs must be compared to Reporting Limits to ensure they are no greater than the RLs.

5.2.3 It is the responsibility of the area supervisor to obtain the necessary information to update the control limits at a minimum of annually. This may be done in conjunction with QA and the LIMS/MIS department.

5.3 QA Department

5.3.1 It is the responsibility of the QA department to issue a Corrective Action notice to any department who fails to turn in acceptable MDL, IDL, or P&A studies.

5.3.2 It is the responsibility of the QA department to work with supervisors to schedule studies and to maintain files of all current and historical studies.

5.3.3 QA will review and provide the final sign-off that the study meets requirements.

5.3.4 QA will review and provide the final sign-off of reporting limits.

5.3.5 QA will bear the responsibility to maintain the statistically determined control limits and to ensure that they are within those specified in the reference method.

6. Operation procedures

6.1 General

6.1.1 All studies must be given laboratory LIMS ID numbers. Although they may be initially stored in QA, they will eventually be moved into the laboratory filing system and must have identification numbers in order to be able to retrieve the raw data. Identification numbers will almost always be assigned by QA but in the absence of the QA Officer may be assigned by authorization of QA or the Laboratory Director. All studies will use the SAM client code QC_Officer in order to better track them at a later date.

6.2 Instrumental Detection Limits (IDLs)

6.2.1 It is rarely necessary to perform actual IDL studies except for metals analyses. For metals analyses, they are performed quarterly on each instrument. Studies may be useful, however, to demonstrate instrument capabilities and as a tool for estimating the Method Detection Limit (MDL). Although IDLs may be used as estimates to determine appropriate MDL spiking levels, it is strictly prohibited to compute the actual MDLs based on IDL determinations. The following guidelines are provided for several general class of analyses, regardless of whether an IDL is required for that analysis type.

6.2.2 As with all studies, a laboratory ID number should be assigned by QA for tracking purposes. In the case of metals IDLs, the same ID number may be assigned to all of the quarterly IDLs, rather than just one per instrument.

6.2.3 Actual IDLs studies are performed according to the CLP SOW by analyzing 7 replicates of low-level standards made up in the same matrix as all standards and not including any processing steps that would not ordinarily be performed on standards. The levels of those standards should be estimated from manufacturers detection limit specifications.

6.2.4 IDLs should be performed under the same instrumental conditions as will be used to perform actual analyses.

6.2.5 IDL studies must contain the following information (not necessarily in this order) for submittal to QA.

- Laboratory ID number
- Analyst who performed the IDL study
- Instrument name and ID which will distinctly identify that instrument
- Spike level
- Measured concentration of the 7 replicates (per day)
- Standard Deviation

- Mean
- Determined IDL
- Concentration Units
- Date(s) the study was analyzed
- Analysis (i.e. ICP, GFAA, etc.)
- Analysts signature & date signed
- Supervisor or senior analyst review signature & date signed

6.2.6 Spectrophotometry

6.2.6.1 The EPA/CLP SOW for metals requires that the IDL study be run on 3 non-consecutive days at least 7 times each day. It is prepared from an acidified aqueous standard solution made up at 3 to 5 times the manufacturers suggested IDL. The sample standard deviation (n-1) for each individual set of determinations is calculated and the final IDL is calculated as 3 times the average of the standard deviations for the three days. This may be performed using any commercial spreadsheet but care must be taken to insure that it is done using the sample standard deviation (n-1) calculation. For *Microsoft Excel*, this is the =STDEV() calculation. Ten percent of the calculations must be manually verified in order to demonstrate that the spreadsheet calculations are accurate.

6.2.6.2 If other spectrophotometric method IDLs are established by analyzing standards 7 times on 3 non-consecutive days, the calculation of the IDL is performed as described above. In addition, the EPA/CLP method does not prescribe the determination of MDLs. It is standard laboratory procedure to perform an MDL study (see section 6.3) approximately annually for almost all routine methods of analysis, regardless of IDL frequency or other determinations.

6.2.7 Chromatography

6.2.7.1 The analyst should use the signal:noise method for determining concentrations to use for an IDL study. A preliminary estimate of 5x signal:noise is to be used; if necessary this will be adjusted and the study repeated.

6.2.8 Gas Chromatography/Mass Spectrophotometry

6.2.8.1 Mass spectral identification criteria are key in selecting target concentrations for the IDL study. The mass spectroscopist's experience in determining the minimum identifiable concentration must weigh heavily in selecting concentrations. All compounds must meet the spectral matching characteristics as called out in the analytical method for the IDL study to be valid.

6.3 Method Detection Limits (MDLs)

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

6.3.2 MDL studies must also be performed when any major changes have been made in an instrument, such as a detector change.

6.3.3 Prior to beginning an MDL study, a laboratory workorder ID must be obtained from QA. The data generated from the study is then referenced to that workorder in the same manner as routine sample data.

6.3.4 MDL studies must contain the following information (not necessarily in this order). This will be accomplished by using the MDL database report plus an MDL Information Sheet (See Appendix 2).

- Laboratory ID number
- Analyst who performed the preparation
- Method number of the preparation (where applicable)
- Date(s) the study was prepared
- Method number of the clean-up (where applicable)
- Analyst who performed the MDL study
- Method number of the analysis
- Date(s) the study was analyzed
- Instrument name and ID which will distinctly identify that instrument; this cannot be a data "channel" from the computer system but must distinctly and uniquely identify that instrument.
- Spike level
- Measured concentration of the 7 replicates
- Standard Deviation
- Mean
- Determined MDL
- Concentration Units
- Reporting Limits (RLs)
- Analysts signature & date signed
- Supervisor or senior analyst review signature & date signed

6.3.5 The analyst must compare the MDLs with their current Reporting Limits (RLs) to ensure that they are no higher than the RLs. In fact, in most cases the MDLs should be demonstrably lower than the RLs unless there is a specific request to report down to the MDL.

6.3.6 If it is determined from the study that the reporting limits must be changed (i.e. the MDL is near to or exceeds the RL and cannot be re-determined with more appropriate results), the QA Officer and the supervisor, often in concert with the Laboratory and/or Technical Director(s), must meet to determine the appropriate course of action. Reporting limits are intended to be at a level for which method precision and accuracy can be obtained. This generally cannot be done when the RL is close to the MDL

6.3.7 In order to determine the Method Detection Limit (MDL), it is first necessary to estimate what the MDL will be in order that the appropriate spiking levels may be used. How this estimate is made is immaterial to the actual MDL determination. Methods for making this determination may include any one or a combination of the following:

- estimating based on the instrument detection limit (IDL) as determined above or by any other means
- estimating based on the previous MDL
- estimating based on 3 times the instrument signal to noise ratio
- estimating based on analyst judgment

6.3.8 A solution is then prepared and spiked into a sample matrix, which is as free as possible of interference and target analytes, at a level that will result in a sample concentration equivalent to 1 to 5 times the estimated MDL.

Note: The CFR states that the recommended concentration levels used to determine the MDL be one to five times the MDL. It later implies that a level of up to 10 times the MDL is acceptable. Although the analyst should make his/her best effort to spike at a level from 1 to 5 times the MDL, Laucks considers up to 10 times the MDL to be a sufficient concentration. Limited exceptions to this rule may be granted as long as the deviations are not great and they are approved by QA.

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

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

6.3.9 Preparation of Spiked Samples

6.3.9.1 The MDL is almost always determined in reagent water or clean sand. Prepare a laboratory standard containing all analytes of interest at a concentration which is at least equal to or in the same concentration range as the estimated MDL. The analyte concentration should not exceed 5x the estimated MDL but allowances may be made up to 10x the determined MDL.

6.3.9.2 It is extremely rare that Laucks will perform studies for other than reagent water or soil. Soil matrix will almost always be represented by clean blank sand except for metals analyses where even clean sand contains levels of some metals which exceed the 10x acceptance criteria. For such analyses, reagent spikes are used containing only the digestion/preparation reagents. MDLs on other matrices will generally only be performed upon specific client request.

6.3.10 Calculation of recovery statistics

Note: All values are used without correcting for native concentration. As previously mentioned, if blank correction is a part of the method, the average blank value is used for correcting analyte concentration measurements. In almost all methods, however, blank correction is forbidden.

6.3.10.1 The sample standard deviation is calculated as follows:

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

where:
s is the standard deviation estimated with n-1 degrees of freedom.
X_i = sample value for replicate *i*
$\overline{X}$ = mean of all of the replicates
n is the number of replicates

6.3.10.2 The Student's *t* statistic is determined for (*n* -1) degrees of freedom at the 99% confidence interval (CI). A Student's *t* table for the 99% CI is provided in Appendix 1. For most data sets, using n=7 sample readings, the *t* value is 3.143.

Note: In some cases, it may be determined that it is useful to prepare an additional sample so that, in case of laboratory accident, at least 7 are available for statistical analysis. Whether or not this is done, all samples analyzed **must** be used in the statistical evaluation unless there is a strong reason to reject one or more of the data sets, such as obvious contamination, abnormally poor surrogate recovery, set of values that are in obvious and significant disagreement with all of the others, or spilled sample. It is inappropriate to reject data which do not have an overriding reason to do so. The reason for rejection must be clearly documented in the data file. If more than 7 points are used in the MDL determination, the current MDL database will not accommodate the calculation. In this case, the determinations will necessarily be done using a spreadsheet program.

6.3.10.3 The MDL determination then becomes:

$$MDL = t_{99\%CI} * s$$

where:
$t_{99\%CI}$ = the Student's t value at the 99% confidence interval
s = the sample standard deviation as calculated above

6.3.10.4 The MDL, standard deviation and Student's t statistic for the appropriate number of replicates at the 99% CI are automatically calculated when using the Laucks MDL database.

6.3.11 Methodology Exceptions/Specifics

6.3.11.1 Wet Chemistry

6.3.11.2 The MDL for all titrimetric determinations is set as the value determined by 0.2 ml of titrant at the method specified titrant strength and sample aliquot size. This would include all tests such as versenate hardness, alkalinity, argentometric or mercurimetric chloride, titrimetric COD, etc. Karl-Fisher moistures would be an exception to this; the MDL is taken to the value determined by 0.05 ml of titrant, the method specified titrant strength, and sample size.

6.3.11.2.1 The MDL for all gravimetric residue determinations (total solids, total suspended solids, etc.) is set as the value determined by a weighing of 0.2 mg at the method specified sample size.

6.3.11.3 GC and Gas Chromatography/Mass Spectophotometry

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

6.3.11.3.2 Likewise, for PCB and other multi-peak GC analyses, pattern recognition may also dictate what can actually be determined. For either situation, analyst interpretation may be in order to confirm actual compound identification. Such interpretation must be noted in the data.

6.4 Reporting Limits

6.4.1 Reporting Limits are generally determined in one of four ways:

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

6.4.2 The administrative decision method is generally based on what the laboratory considers to be a limit which can be obtained on a consistent and reliable basis. Values obtained from statistical determinations of MDLs, for instance, cannot always be confirmed by spectral identification, pattern matching, standard response, or analytical spike recovery. In this instance, the laboratory may choose an RL which is more readily identifiable as a level for which a compound can be so identified and reliably quantified. Administrative decision may also be considered to be a part of the PQL option.

6.4.3 The PQL option is set as a factor times the MDL. This factor may either be set forth in the published method or it may be set by the laboratory. In order to be able to provide consistent and routine reporting limits, the laboratory will generally not reset PQLs when MDLs are re-determined unless the MDL changes by a factor of more than twofold.

6.4.3.1 If it is determined from the study that the reporting limits must be changed (i.e. the MDL is near to or exceeds the RL), the QA Officer and the supervisor, often in concert with the Laboratory Director and/or Technical Director, must meet to determine the appropriate course of action. Reporting limits are intended to be at a level for which reliable identification and reasonably accurate quantitation can be obtained. This generally cannot be done when the RL is close to the MDL.

6.4.4 Project Specific RLs are derived from project requirements and are contractually agreed upon between the laboratory and the client. In any event, the agreed upon limits **cannot** be less than the MDL or IDL.

6.4.5 On occasion, the low standard defines the RL. The decision to use this technique may be any combination of method specific requirements, laboratory decision, or project-specific requirements. In no case will the RL determined from the low standard be lower than the statistically determined MDL.

6.4.6 Reporting Limits are generally detailed in the Detection Limits Database and the LIMS system, unless set by project-specific agreement, in which case they are detailed in documents pertaining to that project and in the ProjQC database. The only persons given the capability to edit the approved limits are QA, LIMS system administrators, and the Technical or Laboratory Director. In most cases, only QA will actually perform any such editing. Note here that the EPA Contract Laboratory Program (CLP) requirements use specific contract required detection limits (CRDLs) or quantitation limits (CRQLs) and any project using the CLP methods will almost always also be reported using the CLP CRDLs or CRQLs. Any exception to the use of the CLP limits in these instances must also be noted in the ProjQC database and on any paperwork defining the details of the project.

6.5 Precision and Accuracy Studies

6.5.1 At a minimum, a one-time demonstration of precision and accuracy (P&A) must be performed for each method.

6.5.2 In some cases, it may also be required that an analyst will be required to perform a P&A study to be considered proficient and capable of independently performing a preparation or analysis.

6.5.3 P&A studies will be performed in accordance with the specific method. Where method-specific performance criteria are not specified, Laucks may choose to set criteria independently. Laucks' criteria, at a minimum, will meet those specified in a given method. Any determination to the contrary must be well documented and in direct consultation with QA and laboratory management.

6.5.4 All P&A studies must be turned in to QA after having undergone supervisory or senior analyst review.

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

- Laboratory ID number
- Analyst who performed the preparation
- Method number of the preparation
- Date(s) the study was prepared
- Analyst who performed the analysis portion of the P&A study
- Method number of the analysis
- Date(s) the study was analyzed
- Instrument name and ID which will distinctly identify that instrument; this cannot be a data "channel" from the computer system but must distinctly and uniquely identify that instrument.
- Spike level
- Measured concentration of the 4 replicates
- Standard Deviation of the recovery tabulated against the published QA Acceptance Criteria Table, where available
- Average recovery tabulated against the published QA Acceptance Criteria Table
- Concentration Units
- Analysts signature & date signed
- Supervisor or senior analyst review signature & date signed
- Raw Data

6.5.6 The mean recovery and acceptance limits must meet the criteria given in the QC Acceptance Criteria Table at the end of each of the determinative methods, when available. Where criteria are not available Laucks may use internal acceptance criteria or defer to a similar technical method with P&A criteria and use this P&A criteria as guidance in establishing performance criteria. In the case of organic SW846 methods, if the criteria are not published in the individual method, the criteria in method 8000 (70%-130%) are followed as a guidance. In many instances, 70-130% is not achievable on a routine basis even by skilled staff. In this case, the laboratory (senior staff in conjunction with QA) may determine its own acceptance limits.

6.5.7 Blank spike analyses are the commonly accepted P&A evaluation. In most methods where criteria are defined, 4 replicates must meet method-specified criteria for the **laboratory** to be considered capable of adequate performance.

6.5.8 The individual **analyst** must be able to analyze four replicates and meet laboratory blank spike control limits to be considered competent to perform the applicable analysis. For purposes of the P&A study, the analyst may be considered qualified if 90% of the analytes in a multi-analyte analysis meet laboratory criteria as long as all analytes meet the default method-specific criteria.

6.5.9 For the laboratory to be able to claim routine performance within specified limits, all analysts performing an analysis must be capable of that level of performance. All analysts must be routinely capable of performance within method-specified criteria and will be evaluated against laboratory criteria, with further action and training in order if they are unable to routinely meet laboratory criteria.

6.6 Control Limits

6.6.1 Initially, when a new method is being implemented or there are insufficient data, the laboratory will use method-specified control limits for evaluation of data. If no such limits exist, the laboratory may elect to use specified limits from a similar method or may set default limits at the laboratory's discretion. These limits may be from the precision and accuracy study for that method. The determination for the suitability of setting any default limits not otherwise specified in a reference method is at the discretion of QA.

6.6.2 During the routine course of analysis, blank spike or laboratory control samples (LCS) and in many cases matrix spikes and matrix spike duplicates (or sample duplicates) will be analyzed. Spiking will occur at the levels specified in the respective methods where available, but will generally be somewhere in the middle of the calibration range.

6.6.3 When sufficient data have been gathered, generally at least 20 data points, the laboratory will undertake the determination of statistically-based control limits. These control limits are based on 3x the standard deviation of recoveries (for accuracy limits) or relative percent differences (for precision limits). In some instances, warning limits may also be established using 2x the appropriate standard deviation.

6.6.4 At a minimum, the control limits will be updated annually on a preparation/analysis/matrix specific basis. The number of data points and spiking levels used to obtain the new limits must be documented when forwarded to QA for approval.

6.6.5 If purchased from a commercial vendor, vendor-supplied control limits for a control sample will be considered adequate for default control limits if they are within the limits specified in the reference method. In addition, if the material is readily available and its composition does not change with every purchase, the laboratory will develop internal limits for that material. These limits may or may not be within the vendor-supplied limits but they **must** be within the method-specified limits.

6.6.6 In general, laboratory determined limits for **control samples** must not exceed method specified limits. If laboratory determined limits do exceed method-specified limits, the entire system must be evaluated to improve method performance. In most instances, it is unacceptable for routine performance to exceed method-specified performance even if the laboratory is using method-specified control limits. This is because even though the

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

6.6.7 The laboratory may also calculate limits for matrix spike and matrix spike duplicate or replicate samples. However, these limits are primarily used to demonstrate method performance on a particular sample or sample-type relative to the routine laboratory sample and exceptions to these limits will generally be allowed as long as control sample limits are met.

6.6.8 The laboratory may be called upon to utilize control limits specified in a method or in a specific contract as designated in the LIMS ProjQC database or supplementary paperwork. The laboratory's overall performance will be considered adequate if internal control limits are within those specified in the reference method. Contractually defined limits will be used for the control samples analyzed under the contract and appropriate corrective actions taken but will not be used as a guide for routine laboratory performance.

6.6.9 For any particular project, if the laboratory exhibits exceptions to the method or contract-specified criteria, appropriate corrective action must be taken. Should routine laboratory control limits be within method or contract-specified criteria, and laboratory limits are exceeded but method or contract limits are met, the data may be reported but should be flagged. Where appropriate, corrective action may still be taken at the discretion of QA.

7. Reports

7.1 Data Package Organization

7.1.1 All work, with the exception of control limit computations, is performed under laboratory workorder ID numbers.

7.1.2 All data supporting the study are provided in a standard format specific to that method. In order to save paper, some items, such as the initial calibration, etc., may be referenced to other workorders. However, it must all be easily recoverable if full documentation is required, up until the standard laboratory data disposal date. Rationalizations for interpreting the results of any study and specific detail which might impact the study should be documented in the file as well.

7.1.2.1 Data files are prefaced with a copy of the summary report containing all of the elements previously noted in this SOP. Where laboratory database reports are available, a copy of the database report must also kept on file by QA. All sign-offs will be handwritten.

8. References

40 CFR Part 136, Appendix B, *Definition and Procedure for the Determination of the Method Detection Limit, Revision 1.11*

EPA Contract Laboratory Program (CLP) Inorganics Statement of Work (SOW), ILM04.0.

EPA "600" Series Methods, section 8.1.1, 40 CFR, Part 136, App. A.

EPA SW846 "8000" Series Methods, both the general method 8000B and the specific methods

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

Appendix I

Student's *t* Values

n	degrees of freedom	*t* value at 99% CI
2	1	31.821
3	2	6.965
4	3	4.541
5	4	3.747
6	5	3.365
7	**6**	**3.143**
8	7	2.998
9	8	2.896
10	9	2.821
11	10	2.764
12	11	2.718
13	12	2.681
14	13	2.650
15	14	2.624
16	15	2.602
17	16	2.583
18	17	2.567
19	18	2.552
20	19	2.539
21	20	2.528
22	21	2.518
23	22	2.508
24	23	2.500
25	24	2.492

Appendix II

MDL Information Sheet

Laucks Testing Laboratories, Inc.
MDL Information Sheet

This form must be submitted in addition to the information supplied on the SAM MDL summary form along with the supporting raw data.

SAM MDL Name: _____

SAM Workorder Number: _____

Analysis:

Analyst: _____

Analysis Method: _____

Analysis Date: _____

Instrument ID: _____

Preparation:

Prepared by: _____

Preparation Method: _____

Preparation Date: _____

Cleanup Method(s): _____

Review:

Analyst's Signature: _____

Supervisor's Signature: _____

QA Approval: _____

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1012

Title: Solvent QC Monitoring for Trace Residue Analysis

Revision history:

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

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

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

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

Controlled Document
No. 20 Assigned to: Tetra

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SOLVENT ACCEPTANCE CRITERIA 8

1. Introduction and Scope

1.1 Description

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

2. Equipment List and Reagents

2.1 Equipment and Reagents

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

3. Safety precautions and Waste Disposal

3.1 Safety Precautions

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

3.2 Waste Disposal

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

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

4. Operation procedures

4.1 Sequestering of Solvent

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

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

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

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

4.2 Initiation, Data Handling and Record Maintenance

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

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

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

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

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

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

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

4.3 Solvent Analysis

4.3.1 Methylene Chloride Acidity

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

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

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

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

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

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

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

4.3.2 Residue Checks

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

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

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

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

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

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

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

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

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

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

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

4.4 Data Package Organization

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

Appendix I

Solvent Acceptance Criteria

Solvent Acceptance Criteria

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

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

Solvent Acceptance Criteria

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

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #: LTL-1013

Title: **Preparation, Storage, Shelf Life and Traceability Documentation of Standards and Reference Materials**

Revision history:

Number	Date
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2	4/17/96
3	6/3/96
4	9/12/00

Controlled Document
No. 20 Assigned to: TETRA

Revised by: Harry Romberg, QA Officer — Date: 9-12-2000

Reviewed by: Monica Carr, Organics Manager — Date: 9/14/00

Reviewed by: Paul Swift, Inorganics Manager — Date: 09/18/00

Approved by: Kathy Kreps, Laboratory Director — Date: 09/20/00

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1. Introduction and Scope

1.1 Method Description

1.1.1 This SOP is intended to describe the way in which standards and reference materials are tracked, prepared, stored and maintained at Laucks, from the time of receipt of the neat or stock materials, solutions or their preparation to the point of use of the working standard. General descriptions of documentation of standard preparation may be present, it is not intended to define the actual method of preparation for each specific method. This is contained in the applicable analytical method SOP. The way in which these standards are tracked, however, is detailed along with the description of storage and shelf life guidance.

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

1.2 Definition of Terms

1.2.1 Standard or Reference Material: these items are defined as any solution of an analyte at a known concentration prepared from purchased neat materials or stock solutions, or from intermediate solutions traceable to purchased materials. This includes calibration standards, independent laboratory control standards (LCS or SRM), spiking solutions, surrogate solutions, independent calibration verification standards.

1.2.2 Shelf Life: The period of time a solution my be held under proper storage conditions before it is no longer considered valid. Materials which have exceeded their respective shelf lives must not be used for quantification purposes.

1.2.3 Expiration Date: The date after which a standard solution is no longer considered valid for quantification purposes. Such a standard is defined as having exceeded its shelf life. Although the expiration date does not either guarantee that a standard is still good if the current date is prior to expiration or that it is bad immediately after expiration, these dates are administrative and must not be exceeded even by 1 day or subsequent data for all but qualitative analyses will most often be considered invalid on this basis alone.

2. Equipment Lists and Standards

2.1 Equipment

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

2.2 Standards

2.2.1 Standards as specified in each analytical SOP.

2.2.2 All standards must also be verified both qualitatively and quantitatively in order to satisfy EPA requirements for traceability. This may be accomplished by either (1) purchasing solutions which have been fully documented by a commercial vendor, or (2) following the recommended steps for traceability as outlined in the CLP Organic statement of work, OLM04.2, Exhibit E, Section 5. *Note: The latter method is not typically used but is allowed if necessary.*

2.3 Standards Logbooks

3. Safety precautions and Waste Disposal

3.1 Safety Precautions

3.1.1 All standards and reference materials including neats or solutions should be handled as if they are hazardous substances.

3.2 Waste Disposal

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

4. Operation Procedures

4.1 Preparation of Organics and Inorganics Materials

4.1.1 General consideration in standard preparations include:

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

4.1.1.2 Choose volumes and aliquots which minimize the number of intermediate dilutions required to obtain final working concentration considering:

- The inherent measurement error, i.e. no aliquots less than 20% of the volume of measurement device whenever possible.
- The ratio of solvent:analyte
- The amount of solution left over for disposal.

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

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

4.1.2 Proper Syringe/Pipette Technique

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

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

4.1.2.3 Always rinse a syringe (organics) at least 3 to 5 times with the appropriate solvent in between measurements, and wipe the syringe with a Kim-wipe.

4.1.2.4 There should be no air bubbles. Either tap them away or discard the solution in the syringe/pipette and obtain another aliquot. Repeat this procedure as often as necessary to remove all bubbles. It may be helpful to use a GC septum with very small (<50 µl) syringes.

4.1.2.5 For organics, when delivering the measured volume to the dilution vessel, fill the vessel 1/2 - 2/3 with the solvent to be used, add the measured aliquot directly into the solvent without touching the sides of the container, and fill to volume with solvent. A sub-surface injection is preferable whenever possible.

4.1.3 When preparing stock solutions from neats, the following steps should be taken.

NOTE: 99.9% of the time, stock standards will be prepared WEIGHT per Volume. DO NOT use Volume measurements for liquids unless EXPRESSLY TOLD to do so by your <u>SUPERVISOR</u>.

4.1.3.1 The dilution vessel (volumetric flask) and stopper should be triple solvent rinsed (last time with the solvent to be used for standard preparation) and allowed to dry completely.

4.1.3.2 The neat is weighed, to 4 significant figures, directly into the volumetric flask and the weight is recorded (to 3 decimal places for volatiles, one less than actually weighed in order to account for possible small losses due to volatilization). Stopper before weighing to avoid compound volatilization if dealing with solvents or volatile materials.

4.1.3.3 For components other than volatiles, the volumetric flask is filled about 3/4 full with dilution solvent and shaken until analyte is completely in solution.

- If the analyte will not dissolve, the stoppered volumetric flask should be sonicated in the sonic bath or thoroughly mixed with a vortex mixer until it does dissolve. (Because sonication heats the solution slightly, the solution should be allowed to cool before dilution to the mark). Consult your supervisor if the compound is not in solution after sonication.
- The volumetric flask is diluted to the mark.
- If the analyte recrystallizes while stored in the refrigerator, the standard should be sonicated before use. Do not aliquot from a cloudy or opaque standard.
- In addition to the normal labeling of the standard, a separate label should be added indicating the need for sonication.

4.1.3.4 For volatiles, the flask is inverted and gently mixed three times after diluting to the mark. The solution in the neck of the flask is then discarded before use.

4.2 Traceability Documentation for Organics and Inorganics Materials

4.2.1 As this SOP is being written, Laucks is transitioning from one standards tracking system which combines some electronic records with a mostly manual paper logbook system to another system which primarily uses an Access database and is incorporated into our ongoing new Lab Information Management system (iLIMS) implementation. This SOP attempts to address both systems since elements of both are currently in place. In some cases, the systems may be both in use concurrently until everyone is assured the new database system meets their needs.

4.2.2 All organic neat standard materials are logged into the new iLIMS database, as described in 4.2.5, when they arrive in the lab. No neat organic material should be used before it has been logged into the database. Inorganic stock materials are currently logged directly into the

appropriate standards logbook but this will change before the next update of this SOP. Examples of some iLIMS standards database screens are provided in Appendix 3.

4.2.3 All standard, spike, or surrogate mixes which are diluted solutions, whether organic or inorganic in nature, are either logged into the database or directly into the appropriate standards logbook. The exact process for any area of the lab is currently changing from the logbook system to the database system and the schedule is different for each area.

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

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

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

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

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

4.2.6 Upon receipt, each purchased neat material, stock, intermediate or working solution is entered into either the database or a standards log-book and assigned a unique LAUCKS

identification number. The information entered in the database or standards logbook must include:

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

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

4.2.7 After each material is logged it is labeled with the LAUCKS ID, date received, date opened (if the material is to be used from the same container more than once) and expiration date (if not already on the label). The accompanying vendor Certificates of Analysis, Purity or Authenticity are labeled with the Laucks ID and filed in a controlled laboratory notebook in the laboratory area. These certificates are then archived through QA when the notebook is full.

4.2.8 Every prepared stock, intermediate or working standard solution is entered into the database or standard log-book and assigned a unique LAUCKS ID number. The logbook entry must include the items detailed in section 4.2.10. The database numbers are determined automatically and do not require this system. Each material must be labeled with:

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

4.2.9 Examples of typical standards logbook entries are provided in Appendix 4.

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

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

4.2.11 All discrete measurements made during a standard preparation must be recorded in the logbook/database, specifically, weights aliquots and final volumes.

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

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

4.2.12 The Laucks internal working material ID must be documented on the manual benchsheet, the analytical run-log or instrument printout to enable tracking back to the parent material. See Appendix 5 for examples of typical bench sheets with standards references.

4.2.13 The iLIMS standards database assigns a unique number using a different nomenclature but the required information about the standards is essentially the same.

4.3 Storage of Standards and Reference Materials

4.3.1 Organic Standards and References Materials

4.3.1.1 Store in vial or bottle which minimizes head space.

4.3.1.2 Use amber or clear glass, screw tops with Teflon-liners when required, and store at, in order of preference, the temperature referenced in the analytical SOP or the temperature detailed below, in the assigned refrigerator.

4.3.1.3 Volatile Standards and Reference Materials

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

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

4.3.1.3.3 Standards are transferred to Mininert vials with Teflon lined septa for daily use and stored in the VOA freezer. When the standards are transferred, the information is recorded in the GC/MS Volatile Standards log book (and/or the iLIMS database).

4.3.1.4 Other Volatile Standard Solutions

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

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

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

4.3.1.4.4 Cover ends of ampules with foil.

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

4.3.1.4.6 Mix gently.

4.3.1.4.7 Partially fill ampules with solution and recap with foil.

4.3.1.4.8 Use CO_2 to cool ampules until crystals form on sides.

4.3.1.4.9 Heat end of ampule with acetylene flame until glass begins to soften.

4.3.1.4.10 Gently pull end until seal is formed.

4.3.1.4.11 Label ampules and store in freezer.

4.3.1.4.12 Record the information in the Mass Spec VOA Standards Log Book (MV) or iLIMS standards database.

4.3.1.4.13 When standard solutions are used they should be transferred to Mininert cap vials with Teflon lined septa properly labeled as previously discussed. The vials are stored in the VOA freezer until discarded.

4.3.1.5 Semivolatile Standards and Reference Materials

4.3.1.5.1 All standards solutions should be stored at a maximum temperature of 4 degrees C (± 2 degrees). Refer to the analytical SOPs for details as some analytes may drop out of solution if at cooler temperatures.

4.3.2 Inorganic Standards and Reference Materials

4.3.2.1 All metals standards are kept in a cabinet in the metals analysis lab. This is at room temperature. Expired standards that are kept for qualitative purposes are kept in the same room, in a different cabinet. These qualitative standards have a special label on the bottles denoting that they are not to be used for quantitative purposes. All other standards are kept at 4°C in a reach-in cooler in the inorganics lab. This cooler is dedicated to standards and SRMs only. No sample storage is allowed in this cooler.

4.4 Shelf Life

4.4.1 Expiration

4.4.1.1 If a parent material has an expiration date of month/year, then the material is considered usable through the end of that month. For example, 01/96, the material expires after 1/31/96. This guidance was obtained from various vendors.

4.4.1.2 All parent expiration dates MUST be entered into the standard log books and/or iLIMS standards database and the expiration date for all resulting child materials must also be entered into the logbook/database and placed on the material label.

4.4.1.3 Note that no child solution may exceed the life of a parent solution or neat material. This stipulation may reduce the shelf life of a prepared solution from that listed in Appendix 2. For instance, if a stock solution is prepared from parent material that has an expiration date of 05/20/95 in 01/95, instead of having a six month shelf life (07/95) the solution will expire, 05/20/95, the same date as the parent.

4.4.1.4 See Appendix 2 for the Table of typical shelf life of standards and reference materials. This table is provided as guidance only. The vendor expiration date (if applicable) and the analytical SOP take precedence over any guidance set forth in the Table.

4.4.1.5 If a standard is past its expiration date it may be used for qualitative purposes only. The standards logbook must be edited to reflect this status and an additional label must be placed on the standard. This label must be bright in color and must clearly indicate that it is to be "Used for Qualitative Purposes Only".

5. Standard Verification

5.1 Criteria

5.1.1 Standards are to have their concentrations verified before use whenever possible. The QC'ing of the standard is to be recorded in the applicable column in the standards logbook unless they are validated in the individual analytical run (such as confirmation by another standard from an independent source). Criteria for standards acceptability are in many cases defined in individual SOPs. In instances where they are not so defined, acceptability criteria compared to an independent source are:

- 80% - 120% for organics
- 90% - 110% for inorganics

Appendix 1

Example Calculations

1. Concentration Calculations from Neat Materials

HELPFUL hint: To keep yourself straight ALWAYS, ALWAYS include the units (mg, ml, etc.) in your calculations.

Example Calculations of Standard Concentrations:

Weight of Neat Material: 0.2500 gm
Volume of Solvent: 10 ml

To Calculate Concentration in mg/L (ppm):

1) Calculation in Steps.

A) $$0.2500gm * \frac{1000mg}{1.0g} = 250mg$$

A.1) Adjust the 250 mg for purity,

i.e. if purity = 90%, 250 mg x 0.9 = 225 mg

If the % purity is ≥ 97%, it is considered 100% pure for standards calculation.

B $$10mls * \frac{1L}{1000mls} = 0.01L$$

C) $$\frac{225mg}{0.01L} = 22500mg / L$$

2) Calculation as a Single Step.

$$\frac{0.2500gm}{10ml} * 0.90(purity) * \frac{1000mg}{1gm} * \frac{1000ml}{1L} = 22500mg / L$$

3)

$$FC = \frac{W}{FV} * P * \text{Conversion Factors}$$

where;

W = Weight of neat material (g)
FV = Final Volume (ml)
P = Purity (%/100)
FC = Final Concentration (mg/L = ppm)

2. Intermediate and Working Standards (Standard Dilution)

$$(FC)(FV) \times 1000 = (AV)(PC)$$

where;

FC: Final Concentration(s) in standard desired. Units=µg/mL.
FV: Final volume of the prepared standard. Units=mL.
1000: Conversion factor from mL to µL
PC: Parent Concentration (standard normally containing high concentrations and is diluted to desired final concentration). Units = µg/mL.
AV: Aliquot Volume of parent standard required to achieve final concentrations desired.
Units: µL (microliter).

a) Neats to Stocks

$$\frac{Purity * 1{,}000{,}000 * W}{FV} = FC$$

where;

1,000,000 = Conversion factor from gram to microgram
W Weight used in standard prep (g)
FV Final Volume (ml)
FC µg/ml = ppm = mg/L

Purity = % Purity/100

For Example: 100% = purity of 1.0
86% = purity of 0.86

If the % purity is $\geq$ 97%, it is considered 100% pure for standards calculation.

Appendix 2

Shelf Life Guidelines

NOTE: *IN NO CASE, will the Laucks' expirations date EXCEED the manufacturer's expiration date.
IN NO CASE, will a child solution have an expiration date that exceeds its parents.

TYPE OF STANDARD	INORGANICS	ORGANICS EXTRACTIONS	ORGANICS INSTRUMENTATION[A]
Purchased Neat	10 Years *[1]	5 Years *[1]	5 Years *[1]
Purchased Stock Solution	12 Months *[2]	12 Months *[2]	6 Months *[2]
Prepared Stock Solution	12 Months	12 Months	6 Months
Intermediate Solution	3 Months	N/A	6 Months
Working Solution	2 weeks *[3]	6 Months	3 Months
Purchased Working Solution		6 Months	3 Months

* 1. Unless the manufacturers expiration date is less than the following, purchased neat standards shelf life will not exceed 10 years materials from the date of receipt for inorganics and 5 years from the date of receipt or 3 years from the date opened for organic materials, whichever is shorter.

*2. Unless manufacturers expiration date is less than the following, purchased stock solutions or intermediates shelf life will not exceed 1 year from the date opened.

* 3. Listed time is maximum. Specific shelf-life criteria are detailed in the individual SOPs.

NOTE: THIS IS A GENERAL PROTOCOL. WHERE POSSIBLE, VERIFY THE INTEGRITY OF THE WORKING STANDARD SOLUTION BY MEETING THE ACCEPTANCE CRITERIA SPECIFIED IN THE ANALYTICAL SOP FROM THE KNOWN TRUE VALUE WHEN ANALYZED AGAINST AN INDEPENDENT LABORATORY CONTROL STANDARD OR A CALIBRATION CURVE.

A. Volatiles:

Method	Expiration Date
CLP OLM04.2	Stock Standards: 6 months or sooner. Gases & reactive compounds: 2 months Secondary standards: 1 month or sooner for gases & reactive compounds, e.g. styrene Other purgeables: 6 months or sooner IS, surrogate & matrix spike: fresh spiking solution weekly or sooner. Calibration standards: weekly or sooner. Standard solutions stored in ampulated glass vials for 2 years from preparation date or shorter if recommended by manufacturer. Once opened, expiration dates above apply. Aqueous standards: 24 hours at 4°C or 1 hour at room temperature; 12 hours if stored on autosampler.
10/92 Low Conc. CLP	Opened stock standards: weekly Aqueous standards: 24 hours. Stock Standards: 6 months or sooner Gases stock standards: 2 months or sooner Secondary dilution standards: 6 months or sooner (gases 1 month or sooner) Working calibration standards: weekly. IS: prepare fresh spiking solution every 3 months or sooner Surrogates: prepare fresh surrogate solution every 6 months or sooner
SW 846 8260B	Stock Standards: 6 months or sooner Gases: weekly if unstable or 6 months if prepared in nitrogen Working solutions: check frequently for degradation or evaporation Calibration standards are prepared daily

Appendix 3

ILIMS Standards Database Screens

The following screens are provided as examples only. Actual use and operation of the database are described in the users guide.

LAUCKS STANDARDS DATABASE Version 1.2

New Std | Edit | Pkg

Current view: all

Std ID: N271 Expired: ☐ Select Std: Copy

Standard Index

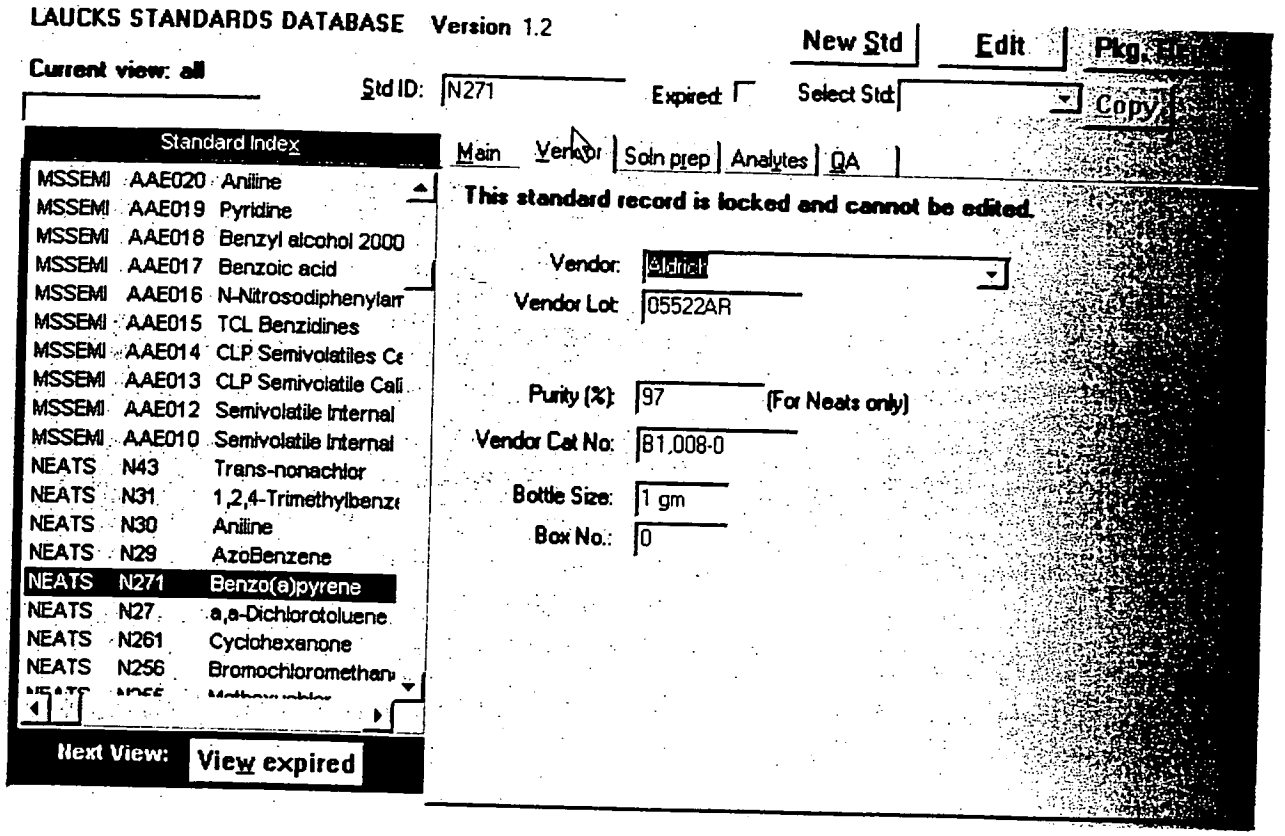
MSSEMI	AAE020	Aniline
MSSEMI	AAE019	Pyridine
MSSEMI	AAE018	Benzyl alcohol 2000
MSSEMI	AAE017	Benzoic acid
MSSEMI	AAE016	N-Nitrosodiphenylam
MSSEMI	AAE015	TCL Benzidines
MSSEMI	AAE014	CLP Semivolatiles Ca
MSSEMI	AAE013	CLP Semivolatile Cali
MSSEMI	AAE012	Semivolatile Internal
MSSEMI	AAE010	Semivolatile Internal
NEATS	N43	Trans-nonachlor
NEATS	N31	1,2,4-Trimethylbenzi
NEATS	N30	Aniline
NEATS	N29	AzoBenzene
NEATS	N271	Benzo(a)pyrene
NEATS	N27	a,a-Dichlorotoluene
NEATS	N261	Cyclohexanone
NEATS	N256	Bromochloromethan

Next View: View expired

Main | Vendor | Soln prep | Analytes | QA

This standard record is locked and cannot be edited.

Type: Neat
Standard Name: Benzo(a)pyrene
Date Prepared (Rcvd): 02/10/1997 Prep by: Aldrich
Expires: 02/10/2002 Logbook No: Neats N271
Dept: NEATS
Solvent:
Solvent Lot:
Final volume: ml
Label Conc.:
QA test date: Tested by: aldrich
QA approve: Aldrich
Comments: Stored at 921
Location: 921
Discarded/Empty: ☐ Discarded Date:


LAUCKS STANDARDS DATABASE Version 1.2
New Std
Edit
Current view: all
Std ID: N271
Expired:
Select Std:
Copy
Standard Index
MSSEMI AAE020 Aniline
MSSEMI AAE019 Pyridine
MSSEMI AAE018 Benzyl alcohol 2000
MSSEMI AAE017 Benzoic acid
MSSEMI AAE016 N-Nitrosodiphenylam
MSSEMI AAE015 TCL Benzidines
MSSEMI AAE014 CLP Semivolatiles Ce
MSSEMI AAE013 CLP Semivolatile Cali
MSSEMI AAE012 Semivolatile Internal
MSSEMI AAE010 Semivolatile Internal
NEATS N43 Trans-nonachlor
NEATS N31 1,2,4-Trimethylbenze
NEATS N30 Aniline
NEATS N29 AzoBenzene
NEATS N271 Benzo(a)pyrene
NEATS N27 a,a-Dichlorotoluene
NEATS N261 Cyclohexanone
NEATS N256 Bromochloromethan
Main
Vendor
Soln prep
Analytes
QA
This standard record is locked and cannot be edited.
Vendor: Aldrich
Vendor Lot: 05522AR
Purity (%): 97
(For Neats only)
Vendor Cat No: B1,008-0
Bottle Size: 1 gm
Box No.: 0
Next View:
View expired

LAUCKS STANDARDS DATABASE Version 1.2

New Std | Edit | Pkg. Report

Current view: all

Std ID: AAE025 Expired: ☑ Select Std: Copy

Standard Index

MSSEMI	AAE032	Semivolatile Internal
MSSEMI	AAE031	Semivolatile Internal
MSSEMI	AAE025	ABN 25
MSSEMI	AAE024	ABN 200 Stock
MSSEMI	AAE023	Semivolatiles Base/
MSSEMI	AAE022	Semivolatile Acids S
MSSEMI	AAE021	EPA CLP SOW OLM
MSSEMI	AAE020	Aniline
MSSEMI	AAE019	Pyridine
MSSEMI	AAE018	Benzyl alcohol 2000
MSSEMI	AAE017	Benzoic acid
MSSEMI	AAE016	N-Nitrosodiphenylam
MSSEMI	AAE015	TCL Benzidines
MSSEMI	AAE014	CLP Semivolatiles Ca
MSSEMI	AAE013	CLP Semivolatile Cali
MSSEMI	AAE012	Semivolatile Internal
MSSEMI	AAE010	Semivolatile Internal
NEATS	N43	Trans-nonachlor

Next View: View expired

Main | Vendor | Soln prep | Analytes | QA

Analytes | Do Calcs

	Stock ID	Amount Used	Units
▶	AAE024	125	uL
	AAE012	10	uL
*			

Record: 1 of 2

Oldest Parent Expiration: 07/14/2000

Dept Filter:

Add by Dept.

LAUCKS STANDARDS DATABASE Version 1.2

New Std | Edit | Pkg

Current view: all

Std ID: AAE025 Expired ☑ Select Std: Copy

Standard Index

MSSEMI	AAE032	Semivolatile Internal
MSSEMI	AAE031	Semivolatile Internal
MSSEMI	AAE025	ABN 25
MSSEMI	AAE024	ABN 200 Stock
MSSEMI	AAE023	Semivolatilles Base/
MSSEMI	AAE022	Semivolatile Acids S
MSSEMI	AAE021	EPA CLP SOW OLM(
MSSEMI	AAE020	Aniline
MSSEMI	AAE019	Pyridine
MSSEMI	AAE018	Benzyl alcohol 2000
MSSEMI	AAE017	Benzoic acid
MSSEMI	AAE016	N-Nitrosodiphenylam
MSSEMI	AAE015	TCL Benzidines
MSSEMI	AAE014	CLP Semivolatiles Ca
MSSEMI	AAE013	CLP Semivolatile Cali
MSSEMI	AAE012	Semivolatile Internal
MSSEMI	AAE010	Semivolatile Internal
NEATS	N43	Trans-nonachlor

Next View: View expired

Main | Vendor | Soln prep | Analytes | QA

Analyte Report

Analyte	Final Conc mg/L
Acenaphthene	25
Acenaphthylene	25
Anthracene	25
Azobenzene	25
Benzo(a)anthracene	25
Benzo(a)pyrene	25
Benzo(b)fluoranthene	25
Benzo(g,h,i)perylene	25
Benzo(k)fluoranthene	25
Butylbenzylphthalate	25
Bis(2-chloroethoxy)methane	25
bis(2-Chloroethyl)ether	25
2,2'-oxybis(1-Chloropropane)	25
Bis(2-ethylhexyl)phthalate	25
4-Bromophenyl phenyl ether	25

Record: 1 of 90

LAUCKS STANDARDS DATABASE Version 1.2

New Std | Edit | Pkg. Report

Current view: all

Std ID: AAE025 Expired ☑ Select Std: Copy

Standard Index

MSSEMI	AAE032	Semivolatile Internal
MSSEMI	AAE031	Semivolatile Internal
MSSEMI	AAE025	ABN 25
MSSEMI	AAE024	ABN 200 Stock
MSSEMI	AAE023	Semivolatilles Base/
MSSEMI	AAE022	Semivolatile Acids S
MSSEMI	AAE021	EPA CLP SOW OLM(
MSSEMI	AAE020	Aniline
MSSEMI	AAE019	Pyridine
MSSEMI	AAE018	Benzyl alcohol 2000
MSSEMI	AAE017	Benzoic acid
MSSEMI	AAE016	N-Nitrosodiphenylam
MSSEMI	AAE015	TCL Benzidines
MSSEMI	AAE014	CLP Semivolatiles Ca
MSSEMI	AAE013	CLP Semivolatile Cali
MSSEMI	AAE012	Semivolatile Internal
MSSEMI	AAE010	Semivolatile Internal
NEATS	N43	Trans-nonachlor

Next View: View expired

Main | Vendor | Soln prep | Analytes | QA

To view a report of the Standards pending QA review

QA Pending

To approve multiple Standards

QA Approve

To view Expired Standards ready to be Discarded

Discard Rpt

Appendix 4

Logbook Examples

LAUCKS TESTING LABORATORIES, INC.
STANDARDS LOGBOOK

#	ANALYTE	ID or STOCK No.	STOCK CONC.	VOL/WT TAKEN	FINAL VOL.	CONC.	SOLVENT	PREP. DATE	INIT.	EXP. DATE	QC'D (Initials)
1	SW846 high std	ME3-96-01 10/19/96	LTL-CAL-1	10 mls	100 mls		HCL 5% HNO3 1% H2O	~~4/3/96~~ KP 4/2/96	KP	7/10/96	KP
		ME4-45-01 4/1/97	LTL-CAL-2	1 ml							
		ME4-32-02 3/14/97	LTL-CAL-3								
		ME3-96-05 7/10/96	1000 ppm Mo 7/10/96								
		ME3-96-09 7/10/96	1000ppm Pb 7/10/96	4 mls							
		ME3-97-08 11/17/96	1000ppm Cu 11/17/96								
		ME3-96-12	1000 ppm Zn								
2	ABN SPE ICP/MS ICV3	ME4-30-01	ICP/MS ICV-1	500 ul	5 mL		10% HNO3 H2O	4/3/96	ABN		
3	AS	ME3-81-9 EXP 5/96	1000ppm	100 µl	5-11-96 ~~10 mL~~ tm 100 mL	1 ppm	5% HNO3	4-3-96	tm	5-96	
4	ICP/MS STD1	ME4-30-02	ICP/MS	50 µl	5 mL		5% HCL H2O	ABN 4/3/96	ABN		
5	STD2		Starch Std	250							
6	STD3			500							

LAUCKS TESTING LABORATORIES, INC.
STANDARDS LOGBOOK

#	ANALYTE	ID or STOCK No.	STOCK CONC.	VOL/WT TAKEN	FINAL VOL.	CONC.	SOLVENT	PREP. DATE	INIT.	EXP. DATE	QC'D (Initials)
MM4-03-04 con't 4	2,4,6-TNT	M-8330-11	1000 PPM	10 ul	50 ml	200 ppb	DI H2O	12/30/95	MH	04/01/96	
⊥	HMX	M-8330-4	⊥	⊥	⊥	⊥	⊥	⊥	⊥	⊥	
01	1,3,5-TNB	M-8330-12	1000 PPM	10 ul	50 ml	200 PPB	Fluid TCLP #1	12/30/95	MH	04/01/96	
	2,4-DNT	M-8330-2									
	RDX	M-8330-5									
	2,4,6-TNT	M-8330-11									
⊥	HMX	M-8330-4					⊥				
02	1,3,5-TNB	M-8330-12					TCLP #2 Fluid				
	2,4,-DNT	M-8330-2									
	RDX	M-8330-5									
	2,4,6-TNT	M-8330-11									
⊥	HMX	M-8330-4	⊥	⊥	⊥	⊥	⊥	⊥	⊥	⊥	

LAUCKS TESTING LABORATORIES, INC.
STANDARDS LOGBOOK

#	ANALYTE	ID or STOCK No.	STOCK CONC.	VOL/WT TAKEN	FINAL VOL.	CONC.	SOLVENT	PREP. DATE	INIT.	EXP. DATE	QC'D (Initials)
1	IC ICV/CCV SK 4/1/96 Cl	ERA 3412	—	—	250ml	129 mg/L	—	logged in 3/18/96	SK	Opened: 4/1/96 Exp: 4/19/97	
	F		—	—		6.47 mg/L	—				
	NO_3		—	—		8.77 mg/L	—				
	SO_4		—	—		11.9 mg/L	—				
2	Cal. Std. F	IC4.84.4	1000 ppb	500 µl	100ml	5 ppb	H_2O	3/20/96	SK	4/3/96	
	Cl	.5	1000 ppb	1000 µl		10 ppb					
	NO_2	.7	100 ppb	2000 µl		2 ppb					
	NO_3	.3	1000 ppb	500 µl		5 ppb ~~10 ppb~~ SK 3/20/96					
	SO_4	.6	1000 ppb	2000 µl		20 ppb					
3	NO_2 ICV	IC4.78.7	100 ppb	1000 µl	100ml	1 ppb	H_2O	3/20/96	SK	3/27/96	
4	NO_2 ICV	IC4.78.7	100 ppb	1000 µl	100ml	1 ppb	H_2O	3/25/96	SK	4/1/96	

Appendix 5

Bench Sheet Examples

Table Name: SW846 Autosampler Type: TYPE TJA
Sample Positions: 257/300 QC Positions: 11/19 # Sets: 1
Probe Station location is rack -1, pos. -1.

--- Racks ---

Rack #	Type	Usage	#Pos Left	Analyses/Pos
1	Aux. (L) Rack	STD/QC/BLANK	11	10
2	Sample (13mm)	Samples	32	1
3	Sample (13mm)	Samples	75	1
4	Sample (13mm)	Samples	75	1
5	Sample (13mm)	Samples	75	1

--- Sample Sets ---

Set#	Type	Prepare?	Description	Method	#Pos	Rack#	StartPos
1	,Y	No	UMC09,UMC11 RE-AS,TL	UMASOIL	43	2	1

--- Preparation Info ---

Report As, Tl
KP 4/10/96

Set#	Uptake	Uptake#2	Final	Dil.Factor

No Samples Prepared.

Rack #1

Pos	Row	Col	Sample Name	Set #	#Used	Type
1	1	1	ICV1 ME4-46-01	-NA-	1	QC Standard
2	1	2	STD4 ME4-46-01	-NA-	1	Standard
3	1	3	STD3 ME4-46-01 5ml/10mls	-NA-	1	Standard
4	1	4	STD2 ME4-46-01 1ml/10mls	-NA-	1	Standard
5	1	5	STD1 ME4-45-02	-NA-	1	Standard
6	1	6	STD0	-NA-	1	Standard
7	1	7	Blank	-NA-	7	Blank
8	1	8	CCV ME4-51-01	-NA-	6	QC Standard

(9...19 Not Used)

Rack #2

Pos	Row	Col	Sample Name	Set #	#Used	Type
1	1	1	CRII1 ME4-45-02	1	-NA-	Sample
2	1	2	ICSABI1 ME4-52-05	1	-NA-	Sample
3	1	3	PBS1	1	-NA-	Sample
4	1	4	LCSS1	1	-NA-	Sample
5	1	5	03040-01	1	-NA-	Sample
6	1	6	03040-01D	1	-NA-	Sample
7	1	7	03040-01S	1	-NA-	Sample
8	1	8	03040-01L	1	-NA-	Sample
[illegible]	1	9	03040-02	1	-NA-	Sample
[illegible]	1	10	03040-03	1	-NA-	Sample
11	1	11	03040-01 5X	1	-NA-	Sample
12	1	12	03040-01D 5X	1	-NA-	Sample
13	1	13	03040-01S 5X	1	-NA-	Sample

LAUCKS TESTING LABORATORIES
ABN GC/MS OPERATIONS LOG

CASE # _____

PAGE 4883-A

3/18/96 IS = MS334-1 DFTPP = MS334-4

RUN #	FILE NAME	CLIENT NAME	CLIENT SAMPLE#	LAB I.D. #	INJECTION INFO	IS A RT	IS A RESPONSE	IS B RT	IS B RESPONSE	IS C RT	IS C RESPONSE
1	MH7501										
2	HC18A	—	SSTD050	MS4-9-1	25. YABN	11.24	55075	14.99	159300	20.44	104485
3	HC18B	—	SSTD200	MS3-89-1	100. X non4/phenol	11.22	59100	14.98	168403	20.42	100469
4	HC180	Kalama	B031396MSVWLA	9603368-BLK	1000→1	11.23	58139	14.98	166207	20.43	99304
5	HC181	Kalama	S031396MSVWLA	—SPK	1000→1	11.24	55723	14.99	155289	20.44	98643
6	HC182	Olympia	—	9603206-ADL	1:4 1000→1	11.24	52364	14.98	158592	20.44	102071
7	HC183	Kalama	—	9603368-01	1:2 100→1	11.24	58997	14.98	168568	20.46	103091
8	HC184	↓	—	-01DL	1:4 100→1	11.24	58386	14.98	173480	20.45	106213

BB 3/19/96

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1017

Title: Internal Audit Procedures

Revision history:

Number	Date
000	5/13/93
001	3/3/96

Written by: Harry Romberg Date: 3/3/96
Harry Romberg

Approved by: Karen T. Kotz Date: 3/3/96
Karen T. Kotz, Laboratory Director

UNCONTROLLED

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1. Introduction and Scope

1.1. Method Description

1.1.1. The purpose of this procedure is to provide instructions for planning, performing and reporting QA/QC audits within the laboratory.

1.1.2. This method is restricted to use by, or under the supervision of personnel experienced in the technique described.

1.2. Discussion

An Audit of the facility is performed for the following reasons:

1.2.1 To determine that contractual and regulatory obligations are fulfilled.

1.2.2 To determine that procedures and standards are being followed, and to insure good laboratory practice. These audits will include, but are not limited to the refrigeration unit temperatures, logbooks, balance calibrations, data, and standards traceability.

1.2.3 To establish that quality assurance objectives are met, including holding times, use of approved analytical methods, and stated objectives for precision and accuracy.

1.2.4 To serve as a management tool to evaluate appropriateness of quality assurance policies.

1.2.5 To identify potential or actual deficiencies for the purposes of evaluating compliance with requirements and providing the means for correction.

1.2.6 To determine that records are prepared and maintained as required.

1.3 Documentation and Frequency

Documentation required is specified in the text and the frequency shall be as required by the QA Manager, but at least one technical audit shall be performed annually for each department. This audit may take place in parts, with additional and more extensive audits being scheduled as deemed necessary.

1.4. Definition of Terms

1.4.3 This section defines terms and acronyms as they are used in this SOP.

1.4.4 SOP: Standard Operating Procedure

1.4.5 QA: Quality Assurance

1.4.6 QC: Quality Control

1.4.7 Audit: A planned and documented activity performed to determine by investigation, examination, or evaluation of objective evidence the adequacy of and compliance with established procedure, instruction, and other applicable documents and the effectiveness of implementation. An audit should not be confused with surveillance or inspection activities performed for the sole purpose of process control or product acceptance.

1.4.8 Auditor: Any individual: who performs or assists in the performance of any part of an audit, including technical specialists.

1.4.9 Lead Auditor: An individual who is qualified to organize and direct an audit, report audit findings, and evaluate proposed corrective actions.

1.4.10 Finding: Departure from approved procedures, program requirements, or other applicable documents that have, or in the immediate future could reasonably be expected to have, an adverse effect on the adequacy or effective implementation of the Laucks QA program. This would be ranked as a **critical** discrepancy in the audit report.

1.4.11 Deficiency: Departure from approved procedures, program requirements, other applicable documents, or good management practices that, if not corrected in a timely manner, could reasonably be expected to have a future adverse effect on the adequacy or effective implementation of the Laucks QA program. This would be ranked as a **minor** discrepancy in the audit report.

1.4.12 Discrepancy: Departure from approved procedures, program requirement, or other applicable documents that have, or may have an adverse effect on the adequacy or effective implementation of the Laucks QA program. This includes findings and deficiencies found during the course of an audit.

1.4.13 Recommendation: An observation or advise given to enhance current practices by any individual or department of the Laucks QA program. This would be ranked as a **recommended** item in the audit report.

2. Responsibilities

2.1 It is the responsibility of QA personnel. the auditor and audit leader to perform an audit according to this SOP and complete all documentation required for review.

2.1.1 QA Manager is responsible for the following:

- Approving each detailed audit plan
- Concurring with the adequacy of each audit report
- Issuing the audit report
- Tracking audit status through final closeout

2.1.2 If an audit team is used. the following responsibilities fall upon the Audit Team Leader. If an audit team is not used. the following responsibilities fall to the QA Manager:

- Developing the detailed audit plan
- Conducting pre-audit and post-audit conferences
- Supervising the conduct of the audit
- Preparing and signing the audit report

2.1.3 Management of audited departments is responsible for the following:

- Providing reasonable and timely access to personnel. facilities. and records, as required to support the audit process
- Providing timely and adequate response to audit reports. including determination and implementation of corrective actions. as required.
- Verifying initial implementation of corrective action for deficiencies in their areas, if applicable.

2.2 Audits and reports are to be performed by personnel in the laboratory who have demonstrated the ability to evaluate processes in the laboratory with emphasis on Quality Control and Quality Assurance.

2.3 Final review and sign-off of each Audit Finding Report may be performed by either the QA Manager. Lab Director or department supervisor or designee.

3. Safety precautions

3.1. Safety Precautions

3.1.1. Auditors must adhere to the general laboratory health and safety policies during the course of the audit.

3.1.2 Protective eyewear must be worn in all applicable locations at all times during the course of the audit.

4. Calibration and Quality Control

Not applicable.

5. Operation procedures

5.1 General

5.1.1 Audit personnel may be selected and assigned audit responsibilities commensurate with their training and expertise and the special nature of the activities to be audited.

5.1.2 Audit personnel are independent of any direct responsibility for performance of any activity which they will audit. Persons having direct responsibility for performance of the activities are not involved in the selection of an audit team.

5.1.3 Audit team members shall have received appropriate indoctrination and training for auditing.

5.2 Audit Planning

5.2.1 The QA Manager. or designee shall develop an audit plan which shall be the basis for the audit. The audit plan is documented on Audit Plan Form (Se Appendix I).

5.2.2 The QA Manager shall develop an audit checklist appropriate to the activity or area being audited. The checklist should contain auditable requirements extracted from the QA Manual,

applicable SOP's or guidance documents, such as EPA SW846. Checklists are designed for each Department by the QA Manager and can be accessed by the QA Department.

5.2.3 The QA Manager shall ensure that the checklist provides an adequate means for indicating whether the question is satisfactorily answered.

5.2.4 Audits are scheduled in a manner to provide coverage and coordination with ongoing QA program activities.

5.2.5 Audits are scheduled at a frequency commensurate with the status and importance of the activity. Within the audit program, each department of the laboratory and each element of the Laucks-QA program is audited, at a minimum, at least once annually.

5.2.6 The QA Manager notifies the audited department, in writing, prior to the audit to provide the subject and scope of the audit, audit schedule, and audit team members, if applicable.

5.3 Audit Performance

5.3.1 The QA Manager and (when required) the appointed audit team members shall proceed through the audit checklist recording evidence of compliance, discrepancies, or recommendations.

5.3.2 During the audit, the QA Manager or appointed team member shall use their best judgment to determine if there is a need to audit at a greater depth than the checklist indicates. If this is the case, the checklist shall be modified accordingly.

5.3.4 Objective evidence is examined, and essential information is recorded, such as the identification of specific evidence examined, specific details of discrepancies or adverse conditions, and applicable references.

5.3.5 The QA Manager shall identify each finding, deficiency, or recommendation in a QA audit report. Findings, deficiencies and recommendations will be listed by department and sequentially numbered in the QA audit report.

5.4 Audit Report

5.4.1 The QA Manager or his designee shall prepare an audit report which should address the following:

5.4.1.1 Date and location (Laucks-department) of the audit.

5.4.1.2 Purpose and scope of the audit.

5.4.1.3 Audit team members (when applicable) and the people contacted during the audit.

5.4.1.4 Description of items. including the rank. type and detail of the audit finding requiring corrective action. The description of the items must be in sufficient detail to enable investigation. evaluation. and correction of the finding. (See Appendix II - Audit Finding Report Form) The report may also include the area affected (See Table in Appendix III) and Finding Type (See Table in Appendix IV)

5.4.1.5 Due date for completion of corrective action plans.

5.4.2 The QA Manager shall issue the audit report to the appropriate levels of Laucks management within four following the audit. This report shall include a copy of each finding. deficiency and/or recommendation.

5.5 Audit Closure and Follow-Up

5.5.1 The appropriate Laucks Management (departmental supervisors, laboratory director hall investigate the reported finding. deficiency or recommendation and do the following:

5.5.1.1 Determine the actions required to correct the discrepancy.

5.5.1.2 Evaluate each discrepancy to determine the root cause of the problem and any generic implications.

5.5.1.3 Determine the corrective action required to correct the discrepancy and to prevent recurrence.

5.5.1.4 Document corrective action and indicate corrective action commitment date.

5.5.1.5 Sign. date. and return the completed form to the QA Manager within the assigned time frame given in the audit report.

5.5.2 The QA Manager shall evaluate each discrepancy/recommendation response. Inadequate or indeterminate responses shall be returned for reexamination of the problem and revised corrective action.

5.5.3 The QA Manager shall verify the corrective action, as stated in the response, and make sure it has been implemented and accomplished as scheduled.

5.5.4 An interim status report of corrective action completion may be issued

5.5.5 After verification of corrective action, the QA Manager shall issue a report stating that all corrective action has been completed and the audit is closed.

5.5.6 If a stalemate is reached concerning either the validity or resolution of an audit finding, affected personnel escalate the concern to the appropriate level of management to effect a resolution.

5.6 Records

The QA Manager shall ensure that the following audit documentation is maintained on file:

5.6.1 Completed audit checklist.

5.6.2 Audit Report (includes findings, deficiencies and recommendations).

5.6.3 Corrective Action (response to discrepancies).

5.6.4 Records pertaining to the completion of corrective action.

5.7 Audit Discrepancy Tracking

5.7.1 Audit discrepancies will be categorized to facilitate tracking and trending of recurrent problems. The categories are as follows:

- Logbook Maintenance
- Document Control Procedures
- QC Procedures
- Standard Operating/Quality Assurance Procedure
- Analytical Method
- Purchasing/Procurement Document Control
- Standards Preparation/Documentation
- Safety/Reagent Labeling or Storage

- Training/Records
- Good Laboratory Practices
- Other

5.7.2 Explanations of Categories Listed Above

5.7.2.1 Logbook maintenance findings include but are not limited to the following: logbooks not being maintained in accordance with Laucks policy, improper entries into logbooks, improper error corrections, logbooks not being kept up to date.

5.7.2.2 Document Control Procedure findings include but are not limited to the following: documents being maintained in such a way that is non-complaint with Laucks document control procedures (this includes archives, SOPs, QAPs, Chemical Hygiene Plan, HTVRs, and forms), records being stored in work areas for longer than 6 months, improper handling of controlled procedures.

5.7.2.3 QC procedure finding include but are not limited to the following: temperatures of ovens and refrigeration units not being monitored in accordance with procedures, balances and pipettes not being verified as required.

5.7.2.4 Standard Operating Procedure and Quality Assurance procedure findings include any case where a procedure has not been followed in full and has not been documented on the applicable corrective action from.

5.7.2.5 Analytical methods findings involve cases where the approved and required analytical method has not been followed to the full extent and there is no documentation that communicates this.

5.7.2.6 Purchasing and procurement document control findings involve instances where the appropriate procedures have not been followed in full. This type of finding includes but is not limited to the following: un-approved use of standards or solvents, lack of certification documentation, etc.

5.7.2.7 Findings for standards preparation and standards documentation include but are not limited to the following circumstances: improperly prepared standards, improperly documented standard preparation, inadequate verification documentation, lack of documentation when procedures are not followed in full.

5.7.2.8 Safety and reagent/chemical labeling findings involve any deviation from approved safety and waste procedures and the chemical hygiene plan.

5.7.2.9 Training and training records findings involve lack of training records, and personnel performing analysis without appropriate qualification documentation.

5.7.2.10 Good Laboratory Practice findings involve significant figures, temperature monitoring, calibration techniques and other associated activities involved with safe and accurate laboratory practices.

6.1 References

Laucks Quality Assurance Plan

Applicable SOPs

Audit Database Tables

Appendix I

Audit Plan Form

LAUCKS Testing Laboratories

Audit Plan

Area to be Audited: _____

Lead Auditor: _____

Audit Team Members (if applicable): _____
_____

Date of Audit: _____

Type of Audit: _____

Checklist(s) to be Used: _____
_____
_____
_____

Individuals Contacted During the Audit: _____
_____
_____
_____

Audit Debrief Date: _____

Report Issued Date: _____

Signature of Lead Auditor: _____

Signature(s) of Team Members: _____
_____
_____

Appendix II

Audit Finding Report Form

Audit Finding Report

Audit Number: Example	Finding Number 1
Facility:	Audit Date:
Auditing Body:	Audit Type:
Lead Auditor:	Affected Area: GC-Semivolatiles

Related Findings:

Finding Rank: Minor Repeat Finding?: No

Finding:

Corrective Action Response:

Opened By:	Date Opened:
Response By:	Response Date:
Corrective Action By:	Scheduled Completion Date:
Verified By:	Date Verified:

Appendix III

Area Names

Depart	Department	DepartmentDescription	Sup ID #
ARCH	Archive	Archive of Documents in QA	0006
BP	Bottle Prep	Bottle Prep	0008
DM	Data Management	Data Management and Administrati	0008
EXT	Extractions	Extractions	0027
GCEF	GC-Extractable Fuels	Extractable Fuels by GC/FID	0038
GCS	GC-Semivolatiles	GC-Semivolatiles	0048
GCV	GC-Volatiles	GC-Volatiles	0038
MSS	GC/MS-Semivolatiles	GCMS-Semivolatile	0048
MSB	GC/MS-Semivolatiles & Volatile	GC/MS-Semivolatiles and Volatiles	
MSV	GC/MS-Volatiles	GCMS-Volatile	0038
SAF	Health and Safety	Health and Safety	0006
HPL	HPLC	HPLC	0038
IN	Inorganics	Metals and Wet Chemistry Office	0053
MIS	LIMS and MIS	LIMs and MIS	0070
MET	Metals	Metals and Metals Prep	0067
MTI	Metals Instrumentation	Metals Instrumentation	0067
MTP	Metals Preparation	Metals Preparation	0067
PM	Project Management	Project Management	0008
QA	Quality Assurance	Quality Assurance	0006
SM	Sales and Marketing	Sales Department	
SC	Sample Control	Sample Control	0008
SP	Special Chemistry	Special Chemistry	0053
WC	Wet Chemistry	Wet Chemistry	0053
YAK	Yakima Office	Yakima Office	0072

Appendix IV

Finding Type

ID of Finding Type	Finding Type
BA1	Balance - Not Certified Annually
BA2	Balance - Not Checked Daily With Class S Weights or as used
BA3	Balance - Weights Not Certified Annually
BA4	Balances - Weights used for calibration do not correspond to weights used for analysis
CA1	Corrective Action - Procedures Not Developed
CA2	Corrective Action - NVC Not Being Tracked
DL1	Documentation/Logbooks - Error and Corrections not be documented correctly
DL2	Documentation/Logbooks - incomplete columns, not properly bound
DL3	Documentation/Logbooks - Not Maintained or used
DL4	Documentation/Logbooks - Inadequate Review
DR1	Data Review - Not Being Performed
DR2	Data Review - Not Being Documented
DR3	Data Review - No SOP
DR4	Data Review - No QC Decision Matrix Available
EC1	Electronic Backup - Not Being Performed
EC2	Electronic Backup - Not Inventoried For Retrieval
GL1	Good lab practice - misc GLPitems
MD1	Methods - No SOP/cribsheet available at time of audit
MD2	Methods - SOP/Cribsheet in use not current controlled version
MD3	Method- controlled SOP/Cribsheet is not being followed or doesn't match current practice
MD4	Methods - The controlled SOP is Non-compliant with the referenced published method
MD5	Methods - SOP/Crib sheets in use & not controlled, meaning draft or handwritten SOPs in use
PE1	Performance Evaluation Samples - Results are outside warning limits, check for error
PE2	Performance Evaluation Samples - Results are outside control limits, not acceptable
PE3	Performance Evaluation Samples - Results included misidentified compounds, not acceptable
QA1	QA - QAP/SOP Document Control Not in Place or Used
QA2	QA - Precision and Accuracy Data Not Current
QA3	QA - MDL/IDL Not Current
QA4	QA - QC Limits Not Determined or Maintained
QA5	QA - Control Charts Not Developed or Maintained
QP1	QAPlan - No QAP Available
QP2	QAPlan - Outdated And Needs Revision
QP3	QAPlan - Has Major Discrepancies With SOPs or practices of the day
RC1	Records Control - Logbooks Not Controlled
RC2	Records Control - Filing not maintained per SOP
RC3	Records Control - No SOPs to desribe System
RC4	Records Control - Not mentioned in QAP
RC5	Records Control - Archiving inadequate
SC1	Sample Control - Building not secured
SC2	Sample Control - COC not established or maintained per client requirements
SC3	Sample Control - Temp/pH not monitored for all regulatory samples
SF1	Safety - No SOP
SF2	Safety - Not Adhering to SOP or Chemical Hygiene Plan
SF3	Safety - Not Adhering to Good Lab Safety Practices
ST1	Stds/Reagents - No SOPs for preparation
ST2	Stds/Rgnts - Prep record inadeq./or not traceable
ST3	Stds/Reagents - Expiration Date Misused
ST4	Stds/Reagents - Not Labeled Properly in the laboratory
SW1	Software - Not Verified and Documented
TH1	Thermometer - NIST Not Available
TH2	Thermometer - NIST Not Evaluated Annually
TH3	Thermometers - Not Calibrated Annually
TH4	Thermometers - Correction Factor Not Applied or misapplied
TH5	Thermometers - Temp. Not Recorded Daily or As Used
TR1	Training - No Formal Program or Documentation
TR2	Training - Incomplete Forms (eg Proficiency, Hrs)
TR3	Training - Not Maintained Consistently

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1018

Title: **Overview of Review and Approval Practices for Validatable Data Packages**

Revision history:

Number	Date
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DATA REVIEW SCHEMATIC 10

1. Introduction and Scope

1.1 Description

1.1.1 This SOP is intended to provide an overview and general organization of data review practices employed for validatable packages. The actual data review processes and check lists specific to those types of analyses are covered in specific SOPs. A schematic diagram of the general review process is provided in Appendix I.

1.1.2 Validatable packages are often similar to the Contract Laboratory Program (CLP) presentation, although the actual analyses themselves and the applicable quality control (QC) may be from SW 846 or other references. If such is the case, the CLP format would be modified to meet the requirements of the referenced methodology. However, the overall review process remains the same.

1.1.3 In-house (non-validatable) data packages receive much of the same review but do not necessarily follow the same process or the same level of documentation. It is not the intent of this SOP to outline the process for these data.

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

2. Equipment List

2.1 Equipment

2.1.1 Data package or the portion of the data package to be validated

2.1.2 SOPs, including associated checklists, for the validation of the analyses of interest

2.1.3 Access to computer programs, etc. which may be required in order to complete the review process

3. Definitions

3.1.1 Validatable Data Package – A complete, self-contained, standardized and organized data package that has adequate information for a reviewer to reconstruct the analytical sequence, calibration and quality control and to verify both the calculations and how well the method functioned for that particular data set.

3.1.2 SOPs – Standard Operating Procedures – written documents that describe a method or operation in adequate detail such that another qualified individual could follow the

outlined procedures with reasonable likelihood of arriving at approximately the same result as another analyst following the same procedure. SOPs contain the steps that the individual must undertake, the sequence of events (analytical sequence) the expected quality control requirements and actions, and assorted other information necessary to repeat the procedure.

4. Responsibilities

4.1 Analyst

4.1.1 It is the responsibility of the analyst to provide the first level of data review and to ensure that all criteria have been met or their failure addressed prior to releasing the data for the next level of review. The analyst may only be the first level of review but is the most important in ensuring that the reported values reflect what was actually present in the samples. It is particularly important that the analyst be proactive in determining any actions that need to be taken in order that they may be completed within the holding time for that analysis and within the turnaround time required by the client.

- The analyst must ensure that the instrument was functioning properly at the time of analysis
- The analyst will ensure that all data comply with the method and project-specific requirements and that any deviations or failures to meet criteria are documented in the project file.
- The analyst must check to see that all calibration criteria were met.
- The analyst must review all quality control data and ensure that criteria were either met or corrective action taken. This action may vary anywhere from simple narration in the report to re-analysis of the sample set, depending upon the QC failure and the method requirements.
- The analyst will review the final data to see that they make sense, that is, the values determined are reasonable, do not disagree with other information the analyst may be aware of, and that the calculated values appear to agree with the raw data.
- The analyst will consult with their supervisor and/or QA to resolve discrepancies with which they are unsure how to proceed.
- The analyst will either transcribe the data into the LIMS or will pass data to the person responsible for transcription in a format which can be easily interpreted.

4.2 Peer or Secondary Review

4.2.1 Data must receive a second level of review from a peer analyst. This analyst should be a person who is familiar with and capable of performing the analysis themselves. If there is no peer analyst available because the analyst in question is the only one experienced with the analysis or for other critical reasons, another qualified individual may substitute for the peer analyst. This person must still be familiar with all aspects of the calculations being performed and the relationships between data and performance of the method in order that the review can be properly conducted. The peer analyst reviewer must:

- Check 100% of the manual entries for transcription errors
- Check 100% of manual calculations for accuracy
- Spot check at least 10% of computer calculations to verify program validity
- Check for completeness of raw data or supporting materials
- Confirm spectral assignments and identification of TICs
- Check for appropriate use of significant figures and rounding
- Check reported values for dilutions
- Check for compliance with Method and project-specific requirements
- Check for reasonableness of data
- Consult with the analyst, supervisor and/or QA to ask questions or resolve discrepancies with which they are unsure how to proceed

4.3 Supervisor

4.3.1 The responsible supervisor or a designated alternate for the area in which the analysis is conducted must provide a technical review of the reported data. This level of review need not be as detailed as the peer review but must include:

- Checking for reasonableness and sensibility of the reported data
- Checking for completeness of the reported information
- Checking for compliance required QC practices including those specified in the Method and those that are project-specific.
- Checking for descriptions of deviations from Method and project-specific QC requirements

- Checking the information in the report narrative for sensibility
- Questioning the analyst or peer reviewer in resolving discrepancies or lack of clarity that may arise and consulting with QA and/or the Lab Director when discrepancies arise with which they are uncertain how to proceed.

4.4 QA Review

4.4.1 QA cursorily reviews most data and periodically, in conducting data audits, reviews select packages more thoroughly. The cursory reviews are generally performed just prior to release of the data. In depth reviews almost always occur after release of the data and are intended more for a review and assessment of laboratory data and processes rather than an assurance of performance on that particular data package. QA may also choose to designate another qualified individual to perform any of these levels of review. Should quality issues arise that have a critical negative impact on the package being thoroughly reviewed, however, QA may call for more specific corrective action. QA may choose to go into any depth in review of data packages, but in general, most reviews will consist of:

- Checking for compliance with required QC practices
- Checking for reasonableness and sensibility of reported data
- Checking for deviations from method or project specific QC requirements
- Checking for compliance with SOPs (periodically)

4.4.2 QA will resolve discrepancies with the supervisor, analyst, or pertinent individual. Some issues may involve corrective actions that require a simple correction while others may require a complete change in routine practices. Documentation of these actions is detailed in the Corrective Actions SOP.

4.5 Project Manager

4.5.1 Project managers do not perform technical reviews but do review case narratives to ensure compliance with contractual agreements. Their responsibilities include:

- Reviewing to ensure that the client requested methodology was used and referenced
- Ensuring that sample entry comments were incorporated, and that concerns that were raised during the course of analysis which required client communication and decisions have been incorporated.
- Reviewing and signing project narratives.

- Reviewing the billing to ensure that the proper invoicing has occurred in conjunction with contractual agreement.

4.6 Management

4.6.1 Senior management reviews case narratives and other components of data packages, should they find cause. They are the parties responsible for approving the release (signing) of reports.

5. Operation Procedures

5.1 General

5.1.1 The processes described below are general. Specific QC and practices, including most of the corrective actions resulting from QC failures are generally described in the appropriate SOPs. The specifics of the review process for individual analyses are specified in the respective data review SOPs along with their associated checklists.

5.1.2 The duties of individuals responsible for various levels of review are specified in the Responsibilities section of this SOP. It is the responsibility of each reviewer to be familiar with this SOP and those specific to their function.

5.2 Corrective Action

5.2.1 Corrective action may be required at any point in the review process. This may include anything from simple recalculation or transcription of a result to complete reanalysis depending upon the circumstance. Many corrective actions are outlined in the applicable SOPs. However, anyone in the review process can either prompt corrective action or, in consultation with any of the others, determine that various actions are either necessary or unnecessary. Corrective actions are individualized to the procedure and circumstance and may be determined to be unnecessary if there is adequate information available to support the reported result. No one in the process should pass along information for which there are unresolved issues.

5.3 Analyst

5.3.1 The analyst must be cognizant of the entire analytical process and document anything out-of-the-ordinary that goes on during the analysis. This may include on-the-spot corrective action, such as dilution and re-analysis. The analyst must also review the data during the production of final results to ensure that all criteria are met and that all appropriate commentary regarding the analysis and any extraordinary steps are clearly noted.

5.3.2 The analyst will then assemble the final data package according to the SOP and submit the data for review to the secondary reviewer. The work of the analyst is the most critical in the review process as this ensures the timely processing of the samples in order to meet holding and turnaround times.

5.3.3 When completed with the data package, the analyst will pass all of the associated materials along to the second reviewer.

5.4 Peer or Secondary Review

5.4.1 The secondary review will usually include use of the checklists associated with the data review SOPs. If in doubt, the secondary reviewer will ask the analyst for further information and not just pass along problems to the next level. In consultation with the supervisor or QA, data may be returned to the analyst for corrective action.

5.4.2 The secondary reviewer will pass the data and checklist along to the supervisor

5.5 Supervisor

5.5.1 The area supervisor or designate will perform the functions outlined under the Responsibilities section, paying special attention to data review checklist items which do not meet method specifications. The supervisor may determine that corrective actions are necessary in the pursuit of data of adequate quality or may consult with QA where the optimal practice is questionable. The supervisor should ensure that corrective actions are all completed and all report commentary is sound prior to submitting the data to the reports department.

5.6 Reporting

5.6.1 The reporting group assembles the respective data packages but bears no responsibility for review other than to ensure that all of the analyses are present in the package, that everyone has input their respective commentary into the report narrative, that all narrative comments have been printed and the appropriate parts of the data package have been assembled. This aspect is detailed in an SOP designed for that purpose.

5.7 Quality Assurance

5.7.1 QA performs cursory reviews of most narratives and data packages before release. QA may call for corrective action at any level should problems be observed which have not been dealt with in an appropriate manner prior to this late stage of reporting. Responsibility to spot and have errors corrected, however, must not be left up to QA if they are spotted earlier or the analysis and reporting of results will almost certainly be

delayed and may even be compromised if reanalysis is required and the delay does not allow them to be completed within holding time.

5.7.2 QA or an approved designee will also perform a more thorough review of select data packages, the scope of which is at the discretion of QA and is not addressed in this SOP. Such review will be more detailed, however, and corrective actions may result which will impact the immediate data or, more likely, affect the processes involved in collecting, reviewing, or reporting data in general.

5.8 Project Management

5.8.1 Project managers review and sign project narratives. They will review only to ensure that the client requested methodology was used and referenced, that sample entry comments were incorporated, and that concerns that were raised during the course of analysis which required client communication and decisions have been incorporated. They must also review the billing to ensure that the proper invoicing has occurred in conjunction with contractual agreement. They may perform these tasks either before or after QA review.

5.9 Management

5.9.1 Management will review and release (sign) narratives.

6. Reports and Documentation

6.1 Data Review and Signatures

6.1.1 Data review forms are provided in individual data review SOPs.

6.1.2 Analyst/reviewer signatures occur on organics cover pages. Inorganics review signatures occur on data cover pages and supervisor signatures are included on both metals and conventional chemistry packages.

6.1.3 Management signatures appear on all final reports.

7. References

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

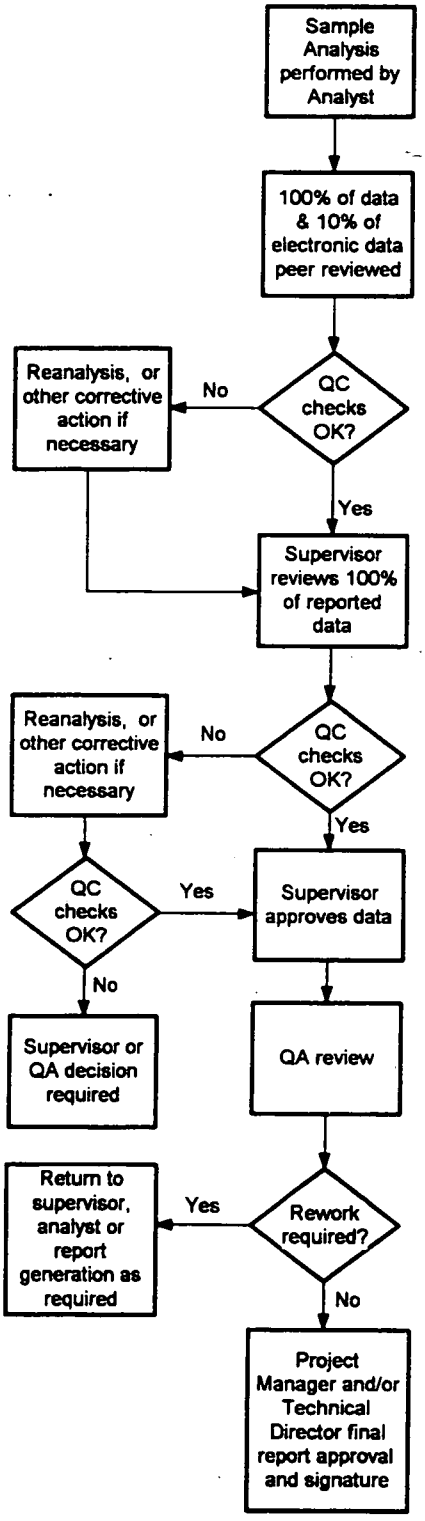
Laucks SOPs
LTL-7001 Metals Data Review
LTL-7002 EPA Inorganics Data Review

LTL-8001	GC Hydrocarbons Data Review
LTL-8002	GC Pesticides/PCBs Data Review
LTL-8003	GC Herbicides Data Review
LTL-8004	GC Volatiles Data Review
LTL-8005	GC Gas/BTEX Data Review
LTL-8201	GC/MS Volatiles Data Review
LTL-8202	GC/MS Semivolatiles Data Review
LTL-8301	HPLC Aromatics 8310 Data Review
LTL-8302	HPLC Ordnance 8330 Data Review
LTL-9001	Inorganic Conventionals Data Review

Appendix I

Data Review Schematic

Data Review and Approval



-100 % manual entries for transcription errors
-100% of manual calculations for accuracy
-10% spot check of computer calculations
-Check for completeness of raw data or supporting materials
-Confirm spectral assignments and identification of TICs
-Check for appropriate use of significant figures and rounding
-Check for compliance with Method or Project QC requirements
-Check for descriptions of deviations from Method or Project QC requirements
-Check reported values for dilutions
-Check reasonableness of data

-Check for reasonableness of reported data
-Check for completeness of the reported information
-Check for compliance with required QC practices
-Check for descriptions of deviations from Method or Project QC requirements
-Check the information for the report narrative

-Check for compliance with required QC practices
-Check for reasonableness of reported data
-Check for descriptions of deviations from Method or Project QC requirements
-Check for compliance with approved SOPs (periodically)

Q:\SOP\METHODS\1000\1018\RVW_CHT1.GFC 6/21/2000

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1019

Title: **Controlling, Maintaining, and Monitoring Laboratory Logbooks**

Revision history:

Number	Date
0	4/11/96
1	5/13/98

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

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

Controlled Document

No. 20 Assigned to: Tetra

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EXAMPLE LOGBOOK REVIEW LABEL 11

1. Introduction and Scope

1.1 Scope

1.1.1 The maintenance of logbooks is essential to monitoring all aspects of laboratory operations including instrument and method performance and in tracking analyses. It is also important to confirming instrument performance at the time of specific analyses and in monitoring ongoing or periodic performance degradation and the steps taken to correct or prevent such occurrences. This document applies to all personnel involved in the preparation, control and use of laboratory notebooks.

1.1.2 More specific instructions for maintaining logbooks can be found in pertinent SOPs, such as LTL-1007 "Maintaining Instrument Records and Logbooks" or LTL-1005 "Analytical Balances" or others specific to other laboratory operations.

1.2 Purpose

1.2.1 The purpose of this SOP is to define the practices used to maintain control and use of laboratory logbooks. This SOP is not intended as a specific description of any particular logbook type but covers the practices that must be in place for all logbooks employed at Laucks.

1.3 Definition of Terms

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

2. Requirements

2.1 Control

2.1.1 Documents that specify quality requirements or activities affecting quality or evidentiary activities shall be controlled to ensure that correct documents are being used and properly archived when completed.

2.2 Maintenance

2.2.1 Quality assurance records (logbooks) shall be compiled and maintained in accordance with approved procedures.

2.3 Monitoring

2.3.1 Logbooks should be periodically monitored to ensure they are being properly maintained and information is being correctly recorded. Standard logbooks and run logs should be monitored at least semiannually by group supervisors or their designees. Maintenance and other logbooks need only be reviewed annually, unless previous review has demonstrated inadequacies in the logbook which require more frequent monitoring.

3. Responsibilities

3.1 Quality Assurance Manager

- Maintain the logs for control of laboratory notebooks and provide control numbers and labels as required.
- Approve format and proposed content of laboratory notebooks; minor changes to pre-printed forms do not need QA approval as long as their basic content does not change.
- Maintain master copies of notebook pages (in instances where pre-printed pages with a specific format are used); this may be in electronic or hardcopy form or both.
- Monitor satisfactory implementation of the requirements of this SOP

3.2 Responsible Supervisor

- Determine the format and content of notebooks used in their respective areas.
- Ensure that QA has been provided with an electronic version of all pre-printed logbook pages in order that they are later available for reprinting or editing.
- Ensure that all laboratory notebooks are properly labeled, including the appropriate control number.
- Ensure that personnel are adequately trained in the proper use of laboratory notebooks
- Periodically review laboratory notebooks to verify satisfactory implementation of the requirements of this SOP. Standard logbooks and run logs should be monitored at least semiannually by group supervisors or their designees. Maintenance and other logbooks need only be reviewed annually, unless previous review has demonstrated inadequacies in the logbook which require more frequent monitoring. This activity may be assigned to another individual but should not be the same individual wh regularly completes the log itself.

3.3 Analyst

- Ensure that they are using the appropriate logbook and understand how to properly fill in the required fields.
- Ensure that any new logbook has been given a logbook number by QA before beginning to use it.
- Ensure that the logbook is clearly identified with an instrument ID and purpose or other appropriate title which will enable the analyst to easily identify the logbook.
- Ensure that if pre-printed logbook pages need to be modified, the modifications are approved by their supervisor and that an electronic copy or, if requested an original hardcopy have been provided to QA.

4. Procedure

4.1 Notebook Structure

4.1.1 Laboratory notebooks may be either bound or unbound as described below. Most logbooks should be bound in some fashion but it is recognized that this is not always possible, such as for vendor service records. These records may be stored in 3-ring binders or other suitable notebooks.

4.1.2 In some instances, logbooks may be created from instrument printouts or other pages that do not lend themselves to being pre-bound. In these instances, the log sheets may be stored in a 3-ring binder or other storage until enough sheets have been accumulated to have them bound with the laboratory comb binder.

4.1.3 All logbooks whether bound or unbound must be controlled by QA as designated by the appropriate QA Book Number label (see example in Appendix 1).

4.1.4 Bound notebooks shall conform to the following:

- Where feasible, binding will be of a type that will make the removal and reinsertion of pages readily noticeable.
- If pre-printed and bound, all pages will be sequentially pre-numbered. If the format of the notebook permits the use of the reverse side of the pages, both sides of each page will contain a sequential page number.

- Each page of the pre-printed bound notebook will contain, as a minimum, the laboratory name, logbook title, and sequential page number. Other elements may also be necessary for any specific logbook.

4.1.5 Unbound notebooks shall conform to the following:

- Unbound pages will be contained in a binder or folder that provides protection from damage.
- Each unbound page will contain a unique identifier (e.g., run number/date). For identification purposes, a continuous printout on fanfold computer paper requires only one identifier unless the sheets are separated.
- As noted above in 4.1.2, some unbound logbooks may eventually be bound if practical.

4.1.6 All notebooks will contain the following information on the cover:

- Laboratory name, Laucks Testing Laboratories, Inc.
- Control number assigned by the Quality Assurance Officer
- The department to which the logbook was issued
- The use of the logbook (i.e. balance calibration, instrument run-log, etc.)
- The department book number or title uniquely identifying that book, as required to identify the specific use of the book. This may include an instrument number or other logbook ID (such as a standards logbook ID). This is in addition to the QA logbook number.
- Start Date, the date on which the first entry was made
- End date, the date on which the last entry was made

4.2 Control of Logbooks

4.2.1 The QA Officer will maintain a master log of laboratory notebooks that contains as a minimum, the following information:

- Unique control number for each logbook
- Logbook title, which should reflect the type of information to be entered.

- Department to whom issued, for accountability only. A logbook will generally be assigned to a work station or function, and in no way is a laboratory notebook to be considered a "personal" notebook.

- Date issued, for accountability only.

- Date closed, for accountability.

4.2.2 Master sheets for each logbook will be maintained by the QA Officer and will be utilized for producing notebooks when required.

4.3 Use of Laboratory Logbooks

4.3.1 The notebook is the basic document for recording information. Entries should be made into the notebook in real time, not written on scratch paper and transferred later.

4.3.2 Handwritten entries should be legible and entered in black or blue indelible ink.

4.3.3 Computer-generated data should be printed out and collected at appropriate times to represent the activities being recorded.

- Computer printouts may be either placed in unbound notebooks as described above, or inserted into bound notebooks.

- Computer printouts or other material inserted into bound notebooks must be securely fastened (tape is preferred) in such a way that removal and insertion of material can be determined readily.

4.3.4 When information from related activities is recorded in more than one notebook, provide adequate cross-reference information in all affected notebooks so that all pertinent data can be readily accessed.

4.3.5 Do not skip pages when entering data. For example, if data is not readily available for entry, do not leave space for later entry. Enter the data when it becomes available and provide adequate cross-references if required.

4.3.6 In cases where partial or complete pages must be left blank and not used, indicate the unused portion by placing a horizontal line at the beginning and end of the unused portion and connecting opposite ends of the horizontal lines with a diagonal, resulting in a Z-shaped figure. The individual striking out the blank area will initial and date the diagonal.

4.3.7 Errors or other changes must be deleted in a similar fashion or with a single-line cross-out which has been initialed and dated. **No erasures, overwriting, white-out or multiple-line cross-outs (blacking out) are acceptable.**

4.3.8 When pre-printed formats are used and all possible entries are not required, the remaining blanks may be struck out with a Z as described above, or entries such as N/A may be placed in the unused blanks.

4.3.9 The individual entering information into the notebook shall initial and date each page used, or in the case of logbooks with ongoing records which do not occupy the entire page, such as maintenance logs or balance logs, each individual entry.

4.4 Supervisory Monitoring of Laboratory Logbooks

4.4.1 Standard logbooks and run logs should be monitored at least semiannually by group supervisors or their designees. Maintenance and other logbooks need only be reviewed annually, unless previous review has demonstrated inadequacies in the logbook which require more frequent monitoring. This activity may be assigned to another individual but should not be the same individual who regularly completes the log itself.

4.4.2 Logbooks should be reviewed using the review items provided in Appendix II, although it is not necessary to actually document the review using this checklist.

4.4.3 Errors should be formally brought to the attention of the responsible individual through the use of Corrective Action Forms. If errors are correctable or items can be corrected for legibility problems, they should be corrected using the proper error correction technique.

4.4.4 Logbooks that have been reviewed are marked with a fluorescent yellow or other colorful label that looks similar to the label in Appendix III.

4.5 QA Monitoring of Laboratory Logbooks

4.5.1 The QA Officer will verify during periodic audit and surveillance activities that notebooks are properly completed and maintained. This will generally be done approximately annually as part of routine audits. This observation does not preclude the requirement for supervisory review.

Appendix I

Example QA Logbook Label

Laucks
Testing Laboratories, Inc.

QA Book No.: _____

Issued To: _____

Used For: _____

Dept. Book No.: _____

Start Date: _____

End Date: _____

Appendix II

Example Logbook Review Items to be Observed

- Have all pertinent fields been filled or marked not applicable (N/A)?
- Has empty space been crossed out properly initialed and dated?
- Have errors been corrected with single-line crossouts, initialed and dated (no obliterations or overwrites)?
- Are all entries clear and easy to read and comprehend?
- If calculations are involved, check several random calculations for error.
- If traceability is involved (as for standards) check several random entries to confirm that the logbook entries can be tracked back to the original entry.
- If standards log, observe some actual standards and compare them against logbook entries for accuracy.
- Are all handwritten entries initialed and dated?
- If the book is beginning to deteriorate, it should be repaired or retired and replaced.

Appendix III

Example Logbook Review Label

Logbook pages _____through_____
have been reviewed for completeness and
spot-checked for accuracy>

Initials:_____ Date:_____

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-2001

Title: **Waste Segregation and Disposal**

Revision history:

Number	Date
1	4/10/91
2	9/11/91
3	4/26/94
4	4/09/97
5	5/12/98

Written by: Harry Romberg Date: 6-2-98
Harry Romberg, Quality Assurance Officer

Approved by: Kathy Kreps Date: 6-2-98
Kathy Kreps, Laboratory Director

Controlled Document

No. 20 **Assigned to:** Tetra

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1. Introduction and Scope

1.1 Method Description

1.1.1 The purpose of this SOP is to describe the laboratory waste disposal scheme currently in place at Laucks. The primary waste streams described include solvents, PCB oil wastes, COD and TOC waste and soil samples. This SOP only covers handling of the waste from the point of collection.

1.1.2 This method is restricted to use by, or under the supervision of analysts experienced in the techniques described. As part of their training for analytical tasks which generate related wastes, each analyst must be trained to properly dispose of the waste or to the consolidate it at the appropriate collection point.

1.1.3 This SOP generally does not cover handling of the waste up to the point of disposal.

2. Equipment List

2.1 Equipment

2.1.1 The equipment necessary to properly dispose of laboratory wastes varies with the type of waste. In general, an appropriate container, packing material, and safety equipment (including clothing, eye wear, and respirators) is required.

3. Safety precautions

3.1 Safety Precautions

3.1.1 Solvent wastes may contain materials flammable at room temperature or lower. Caution should be taken to avoid flames and sparks when in the presence of or handling these wastes.

3.1.2 COD and TOC wastes may contain materials which will burn the skin, eyes, and/or mucous membranes if improperly handled. Precautions should be taken to avoid accidental contact.

3.1.3 All wastes may contain materials which can have both known and unknown long-term health effects. COD and TOC wastes, for instance, contain high levels of mercury salts. Direct contact should be avoided through the use of proper clothing and eye wear, even if no immediate danger is obvious. In the case of volatile solvents and other materials, handling should be done in a well-ventilated area and the exposure to vapors minimized. Where strong fumes are unavoidable, a carbon-filter or other respirator should be worn.

3.1.4 All people who handle waste products or the original reagents should be aware that the laboratory provides safety equipment and has a file containing Material Safety Data Sheets (MSDSs) on all laboratory chemicals in support of OSHA and other safety programs.

4. Operation procedures

4.1 Operations Appropriate to All Collection Areas

4.1.1 Land Disposal Restriction Forms (LDRs), manifests and other paperwork are not extensively discussed in this SOP because the disposal vendor deals with this aspect of the paperwork. It will only be necessary for the person who will be asked by the vendor to sign these forms (usually QA) to check that the information on the forms is accurate and to sign the form.

4.1.2 All waste requiring a Hazardous Waste disposal sticker and manifest will be marked with one of two EPA Hazardous Waste Site numbers. All waste transported from the 921 facility will be numbered WAD981762024 and all waste transported from the 940 facility will be numbered WAD027446608.

4.1.3 The Hazardous Waste Sticker must be labeled with the proper DOT shipping name, even though the disposal company will usually replace the label before shipping. The proper shipping names are listed below in the applicable sections of this SOP.

4.1.4 All collection drums **must** be marked with an appropriately filled out Hazardous Waste sticker (see Appendix A). It is only necessary for Laucks staff to fill in the date that collection was **started** and the contents of the drum in the appropriate space. Hazardous Waste cannot be accumulated for longer than 90 days before it must be disposed. Therefore, do **not** mark the date on the drum until collection is started so as to maximize the allowable time until disposal. This sticker will be replaced by the transporter when they arrive to transport the waste to an approved disposal facility. The replacement sticker will contain all of the information required for transport and disposal.

4.1.5 In addition, corrosive and flammable waste collection drums must have a sticker which indicates their corrosive or flammable nature (see Appendix B).

4.1.6 Once a material has been designated as waste and disposed into the designated drum, that drum must not be stored for longer than 90 days from the point that collection was **started**. This is rarely of concern at Laucks because transport is generally scheduled for most wastes within much less time than the required maximum storage time.

4.1.6.1 The one variation from the above rule is the TOC waste drum. This drum is not a satellite collection point but is actually the catch drum for the waste directly from the instrument. It will be disposed as soon as possible after it has reached capacity.

4.1.7 When collection drums are full or the 90-day limit is approaching, the Quality Assurance (QA) Department must be notified. The preferred lead-time for pickup is 10 working days so QA should actually be contacted 80 days after collection is

initiated. This department, at the time of this writing, is responsible for contacting the appropriate approved transporter and insuring proper disposal takes place.

4.1.8 All questions or concerns regarding hazardous waste operations should first be directed to QA who will determine the appropriate course of action.

4.2 Mixed Solvent Waste

4.2.1 This waste stream is primarily composed of methylene chloride with some acetone and hexane and potentially small quantities of other solvents or dissolved products. The collection point for all of this waste is in the 921 facility (Extractions) solvent locker.

4.2.2 Small, 5 gallon or less containers of other mixed solvent waste may be collected as satellite accumulation units in the inorganics or organics instrument preparation areas but these **must** be transported to the primary drums in Extractions when full. Satellite accumulation containers must be kept closed when not in use and must be marked with the words "Hazardous Waste" or with other words that identify the contents of the container. This will most conveniently be done by using a blank Hazardous Waste Sticker.

4.2.3 When new materials are collected in the primary drum, a Hazardous Waste sticker should be affixed with an initial collection date. The Hazardous Waste stickers should be marked with a DOT shipping name of "**Waste Flammable Liquids**".

4.2.4 Although methylene chloride is non-flammable, other components of these waste drums may be highly flammable. Thus, all of the waste solvent containers must be labeled as flammable.

4.2.5 At least 2 inches of headspace must be left between the top of the liquid and the top of the drum to allow for expansion.

4.2.6 When 3 or more full 55 gallon drums of this waste have been accumulated or 80 days have passed since the beginning of collection of the oldest accumulated drum, QA must be contacted to arrange for transport and disposal.

4.2.7 At the time of this writing, Laucks uses Laidlaw Environmental as the facility of choice for handling this waste stream, although this could be changed at the discretion of QA on either a one-time or ongoing basis.

4.3 Chemical Oxygen Demand (COD) Waste

4.3.1 The primary constituents of this waste are sulfuric acid, water, mercury, silver, and chromium (both tri- and hexavalent). The collection point for this waste is in the inorganics area where CODs are analyzed. These analyses are conducted in small pre-packaged tubes. The reacted tubes are not considered to be waste until they are poured out of the tubes into a collection container.

4.3.2 Collection containers must be labeled with a Hazardous Waste sticker as previously noted. The Hazardous Waste Sticker should be marked with a DOT shipping name of **"Waste Corrosive Liquids, Acidic, Inorganic"**.

4.3.3 In addition to the hazardous waste sticker, these containers should be labeled as corrosive with the appropriate sticker as previously noted.

4.3.4 The waste not held for more than 90 days from initial collection (after pouring from the reaction tubes) until transportation for disposal. After 80 days have passed since the beginning of collection, QA must be contacted to arrange for transport and disposal within the allowable timeframe.

4.3.5 At least 2 inches of headspace must be left between the top of the liquid and the top of the drum to allow for expansion.

4.3.6 At the time of this writing, Laucks uses Laidlaw Environmental as the facility of choice for handling this waste stream. This vendor can be changed at the discretion of QA on either a one-time or ongoing basis.

4.4 Total Organic Carbon (TOC) Waste

4.4.1 The primary constituents of this waste are mercury, potassium persulfate, nitric acid, and water. This waste is collected directly from the instrument into a waste container beneath the instrument.

4.4.2 As this is a continuous process, Laucks does not begin the 90 day clock before disposal is required until this container is full. However, the container must be marked with a corrosive sticker. The Hazardous Waste sticker, in this case, must be dated as soon as the container is full and affixed at that time. The Hazardous Waste Sticker should be marked with a DOT shipping name of **"Waste Corrosive Liquids, Acidic, Inorganic"**.

4.4.3 At least 2 inches of headspace must be left between the top of the liquid and the top of the container to allow for expansion.

4.4.4 As soon as the container is full, the QA department must be notified to arrange for disposal. This waste stream will generally not be held in storage for very long after collection.

4.4.5 At the time of this writing, Laucks uses Laidlaw Environmental as the vendor of choice for handling this waste stream. This vendor can be changed at the discretion of QA on either a one-time or ongoing basis.

4.5 Soil Sample Disposal

4.5.1 State law allows a laboratory to store samples and other materials indefinitely, until they are considered waste and disposed. After that time, from the date of first accumulation, a 90 day timeframe is allowed before disposal must occur. Thus, soils

should not be disposed of until enough have been accumulated to fill at least one 55 gal. drum.

4.5.2 Each drum used for soil waste disposal must be clearly marked with an identifying number which will be used to track which drum contained which samples. When samples are signed-out from their storage areas for disposal, the log sheet must be appropriate marked with the assigned drum number. This will enable the laboratory to track which samples were disposed in which drum.

4.5.2.1 The drums should be marked with a year, location from which they originate, and sequential number. Thus drums for which accumulation began in 1998 from the extractions laboratory would be marked 98-921-01. The -01 being a sequential number that would be incremented with each additional drum -02, -03, etc. throughout 1998. A drum from the main lab would be designated 98-940-01, etc.

4.5.2.2 When samples are transferred from the storage locations to the drums, the Secure Storage Custody Log must be marked to indicate into which drum they were disposed. This should include any bottle identifiers, if necessary to identify just what was disposed. Thus, it will be necessary for personnel disposing of samples to check the drums to make sure there is enough room for the designated samples. Soil samples will generally have their lids removed and disposed in the regular garbage. The jar and all will then be disposed in the waste drum. If the lids themselves contain client identifying marks or locations or have significant amounts of adhering material (oil, etc.) which cannot be readily dumped into the drum the lid will also be disposed into the waste drum.

4.5.2.3 When the drums are disposed, it will be necessary for the laboratory representative who signs the manifest to mark the drum identity on the manifest, although this only needs to be on the laboratory copy if the transporter does not want this information to appear on their copy of the record.

4.5.3 QA must be notified 80 days after accumulation has begun in order to arrange for disposal in a timely manner. If samples are not disposed until there is enough to fill a drum, this timeframe is not of major concern because there are always Hazardous Waste pickups scheduled within any 90 day time period.

4.5.4 The only stickers these drums must have is the Hazardous Waste sticker with the date accumulation was started clearly marked. The Hazardous Waste Sticker should be marked with a DOT shipping name of "**Waste Environmentally Hazardous Substances**".

4.5.5 At the time of this writing, Laucks uses Laidlaw Environmental as the vendor of choice for handling this waste stream, although this could be changed at the discretion of QA on either a one-time or ongoing basis. This vendor incinerates these soils prior

to landfilling which should dispose of any organic materials, including labels, oily material and other hazardous organic substances.

4.6 PCB Oil Waste Disposal

4.6.1 Laucks no longer analyzes many oil samples for PCBs. Thus, this is a very small and infrequent waste stream. However, discussion is presented here in order that there be some documented course of action when it is necessary to dispose of these materials.

4.6.2 All oil samples which are analyzed for PCBs or otherwise known to contain PCBs are treated as PCB oils. No effort is made to distinguish those that actually do contain PCBs.

4.6.3 These oils are accumulated in a 5 gal. drum located in the Extractions laboratory warehouse. This metal drum is stored inside of the lower half of a cut-off plastic 55 gal. drum which fulfills the federal requirements for secondary containment during storage.

4.6.4 When a full drum has been accumulated, Eastern Electric is contacted for pickup and disposal. A signed receipt must be obtained as proof of disposal. Eastern Electric sends a manifest in subsequent mail within 35 days of waste pick-up and must also send a certificate of disposal within 30 days after the actual disposal date.

4.6.5 No annual report to the Department of Ecology is required because the level of PCBs is considered so high as to fall outside of the state's responsibility to monitor. At such levels the federal government regulates the disposal under TSCA. For this reason, it also does not fall within the federal requirement for RCRA governed waste disposal within 90 days. Eastern Electric is responsible for filing appropriate reports. TSCA regulations require that manifests and certificates of disposal be kept on file for a minimum of 3 years.

5. Reports

5.1 Disposal Paperwork

5.1.1 Our current vendor produce all of the required paperwork and insure all of the appropriate container markings (stickers, etc.) are in place prior to shipment. Since Laucks' waste streams are consistent from time to time, our vendors already have the information required to properly fill out the paperwork and Hazardous Waste stickers.

5.1.1.1 The paperwork includes the manifests, land disposal restriction forms and other shipping paperwork. Thus the only requirements of the laboratory are to insure the paperwork is accurate and to sign the appropriate forms.

5.1.2 After the waste has been transported to the disposal or accumulation facility, a signed manifest is returned to the laboratory. This is kept with the permanent record.

5.1.3 All certificates of disposal later provided by the disposal vendor are also associated with any waste shipment and kept with the permanent record.

5.1.4 All records are retained for at least 5 years from the date of shipment of the waste.

5.2 Annual Reporting Requirements

5.2.1 The laboratory must file an annual report with the Washington Department of Ecology (WDOE) for legal and tax purposes. This report is due on March 1 each year. Reports are filed for both the 940 and the 921 facilities (both EPA ID numbers). All waste transported from the 921 facility will be numbered WAD981762024 and all waste transported from the 940 facility will be numbered WAD027446608.

5.2.1.1 The only exception to this reporting requirement is the reporting of the PCB waste oil which is a federally regulated waste and is thus not reported to the WDOE.

5.2.2 The format of this report is defined by WDOE in books provided to the laboratory several months in advance of the due date. Details of this report are not provided in this SOP.

5.2.3 In addition, as part of a WDOE program to reduce hazardous waste in general, Laucks files an annual pollution prevention plan update in September of each year. This report is more loosely defined and the only major requirement is that it be filed. Details of this report are not part of this SOP.

Appendix I

Hazardous Waste Sticker

See directions in this SOP for proper filling out of this sticker.

HAZARDOUS WASTE

FEDERAL LAW PROHIBITS IMPROPER DISPOSAL

IF FOUND, CONTACT THE NEAREST POLICE, OR
PUBLIC SAFETY AUTHORITY, OR THE
U.S. ENVIRONMENTAL PROTECTION AGENCY

PROPER D.O.T.
SHIPPING NAME_____ UN OR NA#_____

GENERATOR INFORMATION:

NAME_____

ADDRESS_____

CITY_____ STATE_____ ZIP_____

EPA
ID NO._____

EPA
WASTE NO._____

ACCUMULATION
START DATE_____

MANIFEST
DOCUMENT NO._____

HANDLE WITH CARE!

CONTAINS HAZARDOUS OR TOXIC WASTES

STYLE WM-6

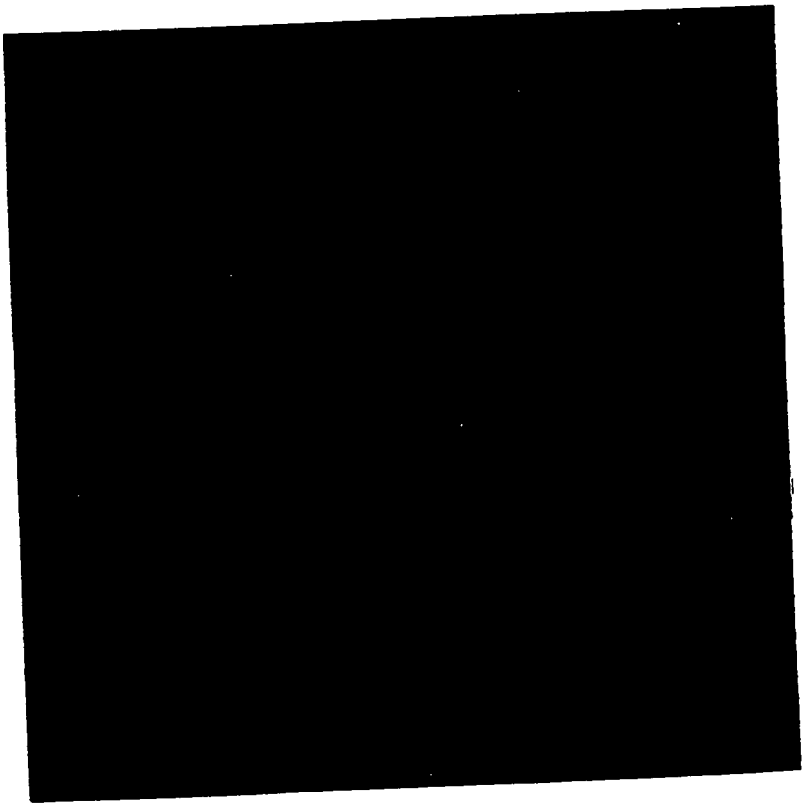
Printed by LABELMASTER, Div. of AMERICAN LABELMARK CO., INC., CHICAGO, IL 60646

Appendix II

Corrosive and Flammable Stickers

8-HML

Published by J. J. KELLER & ASSOCIATES, INC.
Neenah, WI 54956-0368 •(414)722-2848



LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-4002

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

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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
9401499
 KEYS    ST   ORDER # 94-01-496                          RECEIVED 01/12/94   SAMPS
9401485 WR                                               DATE DUE 01/26/94     2
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.

```
Laucks Testing  FRACTION DESCRIPTION 04/05/94 13:36:36
94041
  KEYS                                                    DEPTS/TESTS
9404176-12A   ORDER# 94-04-191/WR CON MARKB
9404176-13A   CLIENT PETER PAN    CAT                                    HIST1
9404176-13B  WRITTEN 04/05/94      BY HOLLYW                             HIST2
9404176-13C
9404176-13D    DASH # 02 A CATEGORY
9404176-14A  SAMPLE ID Mahi Mahi Lot #X3159 (#2)
9404176-15A  COLLECTED 04/05/94          QA/QC   MAT F
9404176-15B     STORED Analyst/Freezer  RECVD 04/05/94 SC   2
9404176-15C                              DUE 04/15/94
9404176-15D  JOB HIST2  FL   QC?        QUOTE    75.00
9404176-16A NAME Histamine               SDG#          StID#
9404176-17A TESTS:DEP/CAT/NAME/D/R
9404176-17B    HISTAM
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, **S**ample **D**elivery **G**roups (SDGs) are commonly created for project work at the time of sample entry. SDGs consist of no more than 20 samples being analyzed for the **same** test. This is in order that the appropriate amount of QC may be analyzed and reported with any sample set. Specifics of the SDG creation process are outlined elsewhere in this SOP.

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

4.2.16 **ALERT:** The 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
CDM      SDG Group  : CDM10        Date Due: 03/13/93        Created:  02/19/93
 KEYS    Fraction   : E SPUMITO                             Updated:  02/19/93
BOGD5G   Project    : CDM (EDB)                 Client:
BOGD5I   SAS Number:                Case Number:          Max. Samps:
BOGD6G
BOGD6I    Work   Samp                                                Associated
BOGD7G    Ord    Num  QC            Client ID          Mat TS pH     Blank
BOGD7I
BRPTSS
CANONS   9302598 17   07-MW-005-0-000-F                 W
CB2SFV   9302598 20   07-MW-007-0-000-F                 W
CB3-3V
CBSFXS
CDM10E
CDM10G
CDM10I
CDM10P
CDM10S
CDM10V
CDM11E
CDM11G
                                                                   CL NL SL J1
5=Lookup,Copy,Print  6=Toggle     F10=More    Shft-Fn=Fn Help
```

SDG, Screen 1

```
                                   SDG Database
CDM      SDG Group  : CDM10        Date Due: 03/13/93        Created:  02/19/93
  KEYS   Fraction   : E SPVMITO                             Updated:  02/19/93
BOGD5G   Project    : CDM (EDB)                 Client:
BOGD5I   SAS Number:                Case Number:          Max. Samps:
BOGD6G
BOGD6I    Work   Samp                 Date
BOGD7G    Ord    Num    VTSR    Collected
BOGD7I
BRPTSS
CANONS   9302598 17  02/16/93 02/13/93
CB2SFV   9302598 20  02/16/93 02/13/93
CB3-3V
CBSFXS
CDM10E
CDM10G
CDM10I
CDM10P
CDM10S
CDM10V
CDM11E
CDM11G
                                                                  CL NL SL J1
5=Lookup,Copy,Print  6=Toggle     F10=More   Shft-Fn=Fn Help
```

SDG, Screen 2

```
                                   SDG Database
CDM      SDG Group  : CDM10        Date Due: 03/13/93        Created:  02/19/93
  KEYS   Fraction   : E SPVMITO                             Updated:  02/19/93
BOGD5G   Project    : CDM (EDB)                 Client:
BOGD5I   SAS Number:                Case Number:          Max. Samps:
BOGD6G
BOGD6I    Work   Samp                          Fractions
BOGD7G    Ord    Num    EDB
BOGD7I
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
COM      SDG Group : CDM10        Date Due: 03/13/93        Created:  02/19/93
  KEYS   Fraction  : E SPUMITO                              Updated:  02/19/93
BOG05G   Project   : CDM (EDB)                Client:
BOG05I   SAS Number:                Case Number:                Max. Samps:
BOG06G
BOG06I
BOG07G
BOG07I
BRPTSS
CANONS
CB2SFV
CB3-3V   Comments:
CBSFXS
CDM10E
CDM10G
CDM10I
CDM10P
CDM10S
CDM10V
CDM11E
CDM11G                                                          CL NL SL J1
5=Lookup,Copy,Print  6=Toggle     F10=More    Shft-Fn=Fn Help
```

SDG, Screen 4

4.6.3 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.

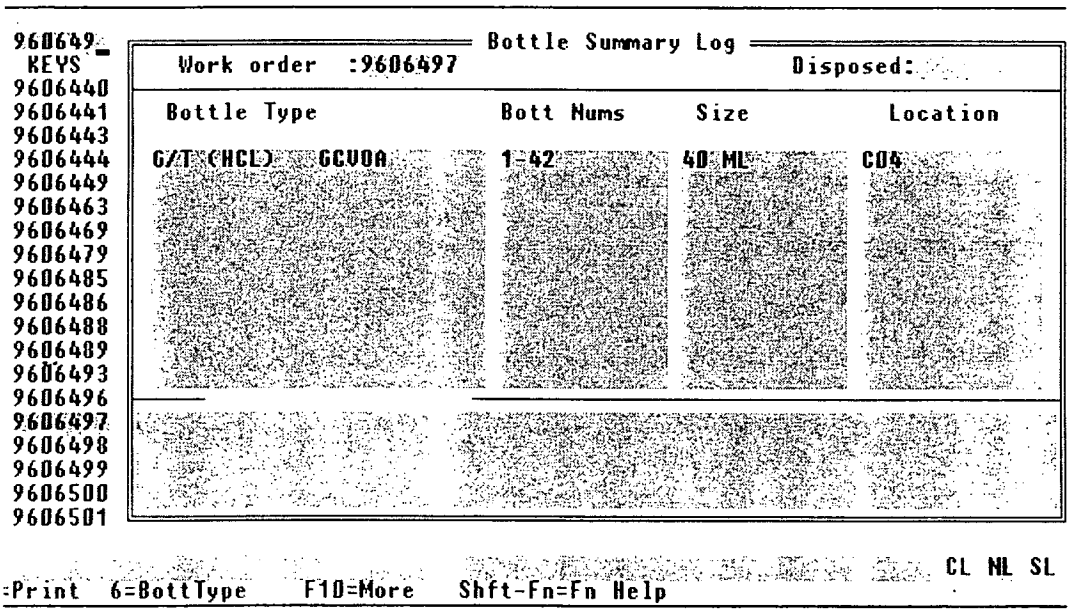
4.8 Storage locations

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

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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:

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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

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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 $\leq$ 2 HCl, no headspace	14 days, with preservation 7 days, if not preserved	Method 602, GC/PID or Method 624, GC/MS
Acrolein and Acrylonitrile	2 - 40 ml containers	Glass, Teflon-lined Septum, 40 ml capacity	Cool, 4° C, if there is presence of residual chlorine then preserve with 0.5 g ascorbic acid, no headspace As above, and pH adjusted to pH 4-5.	7 days 14 days	Method 624, GC/MS
Phenols	1 liter	Glass, Teflon-lined Septum, 1 liter or 1 gallon capacity	Cool, 4° C, if there is presence of residual chlorine then preserve with 0.008% $Na_2S_2O_3$ pH <2 H_2SO_4.	7 days until extraction; 40 days after extraction for analysis	Method 625, GC/MS

A. PRIORITY POLLUTANTS - Organics Analysis (Federal Register Vol. 49, No. 209, October, 1984) (continued)

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Pesticides (Organochlorine Pesticides, and PCB's	1 liter	Glass, Teflon-lined Septum, 1 liter or 1 gallon capacity	Cool, 4° C	7 days until extraction; 40 days after extraction for analysis	Method 608, GC
Polynuclear Aromatic Hydrocarbons (PAHs)	1 liter	Glass, Teflon-lined Septum, 1 liter or 1 gallon capacity	Cool, 4° C	7 days until extraction; 40 days after extraction for analysis	Method 610, GC or Method 625, GC/MS
Base/Neutral and Acid Extractables	1 liter	Glass, Teflon-lined Septum, 1 liter or 1 gallon capacity	Cool, 4° C	7 days until extraction; 40 days after extraction for analysis	Method 625, GC/MS

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

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Halogenated Volatile Organics	2 - 40 ml containers for liquids 20 grams for solids	Glass, Teflon-lined Septum, 40 ml capacity Above or Glass, 2-4 oz. capacity	Cool, 4° C, no headspace Cool, 4° C, packed to avoid headspace	14 days 14 days	Method 8010 GC/Hall - Direct Injection or Headspace, Method 5020 Purge-and-Trap, Method 5030 or Method 8260/Method 8240, GC/MS Purge-and-Trap Method 5030
Nonhalogenated Volatile Organics	2 - 40 ml containers for liquids 20 grams for solids	Glass, Teflon-lined Septum, 40 ml capacity Above or Glass, 2-4 oz. capacity	Cool, 4° C, adjust pH $\leq$ 2 with HCl, no headspace Cool, 4° C, packed to avoid headspace	14 days, with preservation 7 days, if not preserved 14 days	Method 8015 GC/FID Direct Injection or Headspace, Method 5020 Purge-and-Trap, Method 5030 or Method 8260/Method 8240, GC/MS Purge-and-Trap Method 5030

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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	I 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-4103

Title: Reviewing a Sample Delivery Group (SDG) and Updating Projqc in the LIMS

Revision History:

Number	Date
0	4/09/96

Written by: Diana Spence — Date: 4/9/96

Reviewed by: Harry Romberg, QC Officer — Date: 4-9-96

Approved by: Karen J. Kotz, Laboratory Director — Date: 4/9/96

UNCONTROLLED

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1. Introduction and Scope

1.1. Method Description

1.1.1 The project manager (PM) or their designee reviews the LIMS computer entry versus the chain-of-custody (COC) for accuracy. This should be done as soon as practical and within twenty-four hours (24) of sample receipt.

1.1.2 This method is restricted to use by the person (usually a project manager) who performed the work order transmittal.

1.2. Definition of Terms

1.2.1. SDG - Sample Delivery Group

Projqc - Project QC section of the LIMS. This is where all of the work orders for a specific project are listed with additional information about the QC requirements, TATs and deliverables.

2. Equipment List

2.1. Equipment

2.1.1. The person performing this procedure must have access to a computer which is connected to the LIMS.

3. Operation procedures

3.1. Reviewing the SDG Entry in LIMS

3.1.1 Once a work order has been transmitted, go to the SDG section of the LIMS. This is done by entering [SDG, space, SDG name] at the J(1): prompt. The SDG name is found in the fractions sections of the work order in the SDG field labeled 'SDG#'. **Example** - J(1): SDG HCST4. See Appendix I for an example.

3.1.2 In the SDG verify that all the samples have been entered and that the correct 'fraction' of the SDG (I,G, V, P, etc.). The letters refer to the type of analysis, i.e., Inorganics, GC, Volatiles, and PAHs. These are a few of the types of

fractions which might be contained in a SDG. It is not important to know the abbreviations. However, it should be verified that all analyses for each sample can be found in the SDG by looking at the various fractions for that SDG. A fraction may contain more than one analysis. For instance an 'I' fraction (inorganics) might have TOC, Metals and TSS.

3.1.3 When the SDG name is entered at the J(1): prompt, the 'first' page of the first fraction of a SDG will be displayed. The first fraction is determined alphabetically based upon the letter associated with an analysis. For instance, if a work order has analyses for GC analysis (G), metals (I) and PAHs (P), the fractions of a SDG would be listed in LIMS with GC analyses first, metals second, and PAHs last. The information contained on the first page is:

- Laucks sample ID
- QC Designations
- Client ID
- Matrix

3.1.4. Verify that all the samples for that fraction (analysis) have been entered. this is facilitated by entering [F6] which displays the second , third and fourth pages of a fraction. [F6] is a toggle key which pages through the four screens of each SDG entry. The far left hand column of the computer screen will always display the lists of the SDG names with the fractions appendix letter. **Example** - GSI01G (GC fraction), GSI01I (inorganics fractions). See pages 1,2 and 3 of Appendix 1 for examples of each page of a SDG.

3.1.5 The second page of a SDG contains the Laucks samples ID, the VTSR (Verified Time of Sample Receipt) and date collected information.

3.1.6 The third page of a SDG contains the Laucks sample ID and the specific analysis requested. It is important to review this page carefully. If there are many samples in that fraction, they may not all be visible at once. Move down the screen to view all samples by repeatedly pushing the down arrow key, [↓].

3.1.7 Once this fraction has been checked, the other fractions can be checked by using the [F3] (moves the cursor up) and [F4] (moves the cursor down) function keys to place the cursor on the next fraction of the SDG. It is displayed in the column on the left of the screen.. It is possible to view the same page of different fractions by just moving the cursor to the next fraction. For example, if you are in page three of a fraction, when you move to the next fraction, page three of this fraction will

be displayed. **[F6] controls the page selection of the SDG, [F3] and [F4] respectively in the far left column.**

3.1.8 If corrections are necessary, return the COC and log-in documents to the Sample Control department. If the person performing the review makes the corrections, the changes must be saved by entering [F8].

3.1.9 To exit the SDG, enter [F1] until the J(1): prompt is displayed.

3.2 Entering a Work Order into 'Projqc' of LIMS

3.2.1 At the J(1): prompt, enter [projqc, space, projqc name]. Example - J(1): projqc OHM_Hawaii. If you are not sure of the projqc name, a name may be entered which is similar. This will bring up the projqc in the vicinity of the name which is desired. The cursor is moved up, [F3], or down, [F4}, to the desired name. The projqc names will be displayed in the far left column of the screen. See page 1 of Appendix II.

3.2.2 Once the correct project name is displayed, move the cursor down to the next empty line in the work order column. enter the work order number. The SDG will appear in the SDG list column. Move the cursor to the matrix columns (W = Water, S = Soil, O = Oil). Under the appropriate column, enter the number of samples for each matrix. If the SDG is to remain open, no other information should be added. Enter [F8] to save the updated SDG.

3.2.3 It the SDG is to be closed, the due date for the data to be submitted to the reporting department and the due date for the hardcopy report to the client must be entered into the appropriate columns. These dates are displayed in Projqc as 'Office' and 'Client' respectively. See page 1 of Appendix II. Enter [F8] to save these changes.

3.2.4 SDG closure is determined by several factors:

- The number of samples in the SDG, per the EPA definition, should not contain more than twenty samples of the same matrix received over a period of not more than 14 calendar days.

- If it is known that more samples will be arriving for the same project in less than fourteen days, and the SDG is not full, it may be desirable to maintain the SDG in 'open' status until arrival of the next samples.

- If the client desires a fast TAT for the hardcopy package, the SDG should be closed regardless of the number of samples in it.

These are judgment decisions with the exception of the EPA definition of an SDG), which must be made by the project manager.

3.2.5 Once the SDG has been closed, send e-mail notification of the closure to all departments affected. This would be any department involved with the analyses for the work order, Sample Control and the reporting department.

3.2.6 Finally, the paperwork is submitted to the reporting department detailing the nature and status (open or closed) of an SDG. The forms submitted would be the following, arranged in the order listed below:

- Pre-package checklist
- Chain-of-Custody forms (original or top, white copy)
- * Sample Receipt Log (1) CLP (original)
- * Supplemental Sample Receipt Log (original)

* A copy of each of these forms must be made and attached to the copy of the COC for the transmittal.

See Appendix III for examples of these forms.

3.3 Creating a New Projqc

3.3.1 When a new project commences, it may be necessary to create a new entry in Projqc. The factors which would determine if this is necessary may include the following:

- The complexity of the project
- The duration of the project
- How many laboratory departments are affected by the project

Projqc entries are applicable to both CLP and non-CLP project. The information contained in this section of the LIMS is accessible by all laboratory staff who have rights to the LIMS and is a valuable form of communication for project requirements. However, it does not take the place of kick-off meetings for projects or e-mail regarding specific samples problems.

3.3.2 To create a new projqc entry, enter [projqc] at the J(1): prompt. This will display the first projqc entry in the LIMS. The cursor will be in the field called 'Proj Name:'. Enter the name of the new projqc entry. this name may consist of up to ten characters, alpha and numeric, and one underscore in the place of a hyphen, if desired. Once entered, push [Enter]. The cursor will move to the next field, 'Client Code'. See page 1 of Appendix II.

3.3.3 In the 'Client Code' field, enter the correct client code for this work order. This can be found in the work order. Push [Enter]. The cursor will next appear in the 'Desc:' field.

3.3.4 In this field enter a brief description (one cryptic sentence) of the project. Push [Enter]. the cursor will next appear in the bottom line of the projqc header. Enter the correct information for these questions (QAPP?, pH/Temp?). This would usually be 'yes' if the project is CLP or USACE (US Army Corps of Engineers).

3.3.5 Due to software complications, in the field 'Sort by (W/S):' it is best to enter [N].

3.3.6 Enter [F8] to save this new projqc. Item 3.3.1 through 3.3.5 would be considered the header information for a projqc. This is page 1 of the projqc. It is here that the work orders for that project will be entered when the samples arrive at the laboratory.

3.3.7 Next go to page 2 of the projqc. This is accomplished by pushing [F6]. The header information will already be there. The information which must be entered here is:

- Results TAT (Fax)
- Report TAT (hardcopy)
- Deliverables - CLP, LTL (Laucks Testing Laboratory or non-CLP)
- MPR - Monthly Progress Report (required for HAZWRAP projects)
- EDD deliverables - CLP or LTL (any non-CLP, custom EDD)
- Deliverables Comments - Any special project comments
- Penalties - enter any applicable penalties here

Enter [F8] to save this information.

3.3.8 Next go to page three of the projqc. This is accomplished by entering [Ctrl,F6]. of a project can be written as an overview. See page three of Appendix II. The information contained here is:

- Client name
- Project Name
- Overview
- Schedule
- Analytes
- Protocol
- QC
- Turnaround
- Penalties
- CRDLs
- Holding Times
- Deliverables
- Additional Comments

After entering this information, enter [F8] to save it. It may be advantageous to enter [F8] periodically as the information is entered to prevent loss in the event of a power surge.

Appendix I

Example of SDG Entry in LIMS

GSI01
KEYS
GLS08S
GLS08T
GLS08V
GLS09E
GLS09I
GLS09O
GLS09P
GLS09S
GLS09T
GLS09V
GSI01G
GSI01I
GSI01P
GSI01V
GSI02G
GSI02I
GSI02P
GSI02V
GSI03G

SDG Database

SDG Group : GSI01 Date Due: 11/10/95 Created: 10/12/9
Fraction : G SPVMITO Updated: 10/23/9
Project : AFCEE/Chanute 952 Client: GSI (TPH 8015) JP4
PROJQC ID : GS_CHANUTE Max. Samps: 21

Work Ord	Samp Num	QC	Client ID	Mat	TS	pH	Associated Blank
9510423	01		B952/SB-952-1/SS4,6-8/LK	S	88		B1018GSVSLW
9510423	02		B952/SB-952-1/SS9,16-18/LK	S	89		B1018GSVSLW
9510423	03		B952/SB-952-2/SS4,6-8/LK	S	83		B1018GSVSLW
9510423	04		B952/SB-952-2/SS5,8-10/LK	S	84		B1018GSVSLW
9510434	01		B952/SB-952-10/SS8,14-16LK	S	84		B1018GSVSLW
9510434	02		B952/SB-952-3/SS3,4-6/LK	S	84		B1018GSVSLW
9510434	03	MS	B 52/SB-952-3/SS6,10-12/LK	S	89		B1018GSVSLW
9510434	05	BMS	CH-5092500-S-SPOON-ERB-1	W			B1017GSVWLQ
9510434	06		CH-POT-FB-1	W			B1017GSVWLQ
9510434	07		CH-ASTM-5092500-FB-1	W			B1017GSVWLQ
9510459	01		B952/SB-952-4/SS4,6-8/LK	S	84		B1018GSVSLW
9510459	02		B952/SB-952-4/SS7,12-14/LK	S	89		B1018GSVSLW
9510459	03		B952/SB-952-5/SS4,6-8/LK	S	87		B1018GSVSLW
9510459	04		B952/SB-952-5/SS6,10-12/LK	S	87		B1018GSVSLW
9510459	08		B952/SB-952-6/SS4,6-8/LK	S	84		B1018GSVSLW
9510459	09		B952/SB-952-6/SS6,10-12/LK	S	90		B1018GSVSLW
9510497	01		B952/SB-952-7/SS3,4-6/LK	S	89		B1018GSVSLW
9510497	02		B952/SB-952-7/SS8/4-16/LK	S	91		B1018GSVSLW
9510540	01		B952/SB-952-8/SS3,4.0-6.0	S	90		B1018GSV[illegible]
9510540	02		B952/SB-952-8/SS6,10.0-12	S	90		B1018GSVS[illegible]

SDG Database Report
Page 2

Laucks Testing Labs

Report Date: 01/25/96

SDG Group : GSI01 Date Due: 11/10/95
Fraction : G
Project : AFCEE/Chanute 952 Client: GSI (TPH 8015) JP4
SAS Number: _____ Case Number: _____

Work Ord	Samp Num	VTSR	Date Collected
9510423-01		10/11/95	10/10/95
9510423-02		10/11/95	10/10/95
9510423-03		10/11/95	10/10/95
9510423-04		10/11/95	10/10/95
9510434-01		10/12/95	10/11/95
9510434-02		10/12/95	10/11/95
9510434-03		10/12/95	10/11/95
9510434-05		10/12/95	10/10/95
9510434-06		10/12/95	10/10/95
9510434-07		10/12/95	10/10/95
9510459-01		10/13/95	10/11/95
9510459-02		10/13/95	10/11/95
9510459-03		10/13/95	10/11/95
9510459-04		10/13/95	10/11/95
9510459-07		10/13/95	10/12/95
9510459-08		10/13/95	10/12/95
9510459-09		10/13/95	10/12/95
9510497-01		10/14/95	10/12/95
9510497-02		10/14/95	10/12/95
9510540-01		10/17/95	10/15/95
9510540-02		10/17/95	10/15/95

SDG Database Report
Page 3

Laucks Testing Labs Report Date: 01/25/96

SDG Group : GSI01 Date Due: 11/10/95
Fraction : G
Project : AFCEE/Chanute 952 Client: GSI (TPH 8015) JP4
SAS Number: _____ Case Number: _____

Work Ord	Samp Num	JP4	Fractions									
9510423-01		X										
9510423-02		X										
9510423-03		X										
9510423-04		X										
9510434-01		X										
9510434-02		X										
9510434-03		X										
9510434-05		X										
9510434-06		X										
9510434-07		X										
9510459-01		X										
9510459-02		X										
9510459-03		X										
9510459-04		X										
9510459-07		X										
9510459-08		X										
9510459-09		X										
9510497-01		X										
9510497-02		X										
9510540-01		X										
9510540-02		X										

Comments:

SDG Database

G
KEYS
GLS08O
GLS08P
GLS08S
GLS08T
GLS08V
GLS09E
GLS09I
GLS09O
GLS09P
GLS09S
GLS09T
GLS09V
GSI01G
GSI01I
GSI01P
GSI01V
GSI02G
GSI02I
GSI02P

SDG Group : GSI01 Date Due: 11/10/95 Created: 10/12/95
Fraction : I SPVMITO Updated: 10/17/95
Project : AFCEE/Chanute 952 Client: GSI (Inorganic)
PROJQC ID : GS_CHANUTE Max. Samps: 21

Comments:
Use Control F6 for more PROJQC details.

F7=Del,/n,Name 8=Write,/n,In_use 9=Print,Db_enter?,Keys? 10=More,Help,Ke

Appendix II

Example of Projqc in LIMS

OHM
KEYS
HC_MANANN
'C_MANANNS
.IC_MANANNW
KIC_ARCO
LOW_AFB_IN
LOW_AFB_OR
MCCHORDAFB
MCCHORDLTM
METRO_TBT
OHM-HAWAII
OHM-SITEA
OHM-SITEF
PASCO_AIR
PASCO_P
PASCO_S
PASCO_W
Q_CITY_F
SKAGIT
SODASPRING

Project QC Requirements Database

Proj Name: OHM-HAWAII Client Code: OHM_HAWAII Created: 12/19/9
Desc: UST sites Updated: 01/22/9
QAPP? Y pH/Temp? Y Sort by (W/S): N By : DIANA

Ord#	Last Date	SDG#	Office	Client	Matrix W	Matrix S	Matrix O
9512516	12/19/95	HI033	01/31/96	02/02/96	2	3	
9601349	01/22/96	HI034	02/20/96	02/22/96	1	7	
9601351	01/10/96	HI035			3		
9601587	01/22/96	HI034	02/20/96	02/22/96	2	6	

F7=Del,,Name 8=Write,,In_use 9=Print,Help menu,PrtSc 10=More,Help,Key

Project QC Requirements Database

OHM
KEYS
HC_MANANN
HC_MANANNS
HC_MANANNW
KIC_ARCO
LOW_AFB_IN
LOW_AFB_OR
MCCHORDAFB
MCCHORDLTM
METRO_TBT
OHM-HAWAII
OHM-SITEA
OHM-SITEF
PASCO_AIR
PASCO_P
PASCO_S
PASCO_W
Q_CITY_F
SKAGIT
SODASPRING

Proj Name: OHM-HAWAII Client Code: OHM_HAWAII Created: 12/19/9
Desc: UST sites Updated: 01/22/9
QAPP? Y pH/Temp? Y Sort by (W/S): N By : DIANA

Results turnaround 7 WORK.DAYS
Report turnaround 30 CAL.DAYS

Deliverables	Penalties
LTL report	
"CLP" report X	
MPR required	
CLP disk	
LTL disk	
Other	
Other	

Deliverables comments

F7=Del,,Name 8=Write,,In_use 9=Print,Help menu,PrtSc 10=More,Help,Key

File: Y:OHM-HAWAII.PCC
Date: 01/24/96

Client : OHM Remediation Services, Inc.

Project Name: UST Sites in Hawaii

Overview : 90% project consists of Soil samples, 10% water/rinsate samples

Schedule : Delivery of samples over 6 months. Weekly schedule will be obtained one week in advance of sample receipts.

Analytes : Water
29- BTE samples via 8020
36- 8015 Mod (TPH gas or diesel or 418.1)
17- 6010 for Pb, Cd, Cr
30- 8310 Naphthalene, Acenaphthene, Fluoranthene, and low level Benzo(a)pyrene
7- 8080 Low level PCBs 2L to 1mL
7- 8010 tetrachloroethylene, and 111-trichloroethane
10- 1020 flammability
1- HPLC (method supplied by client 2/16) for PGDN and 2-nitrodiphenylamine, Samples to arrive after March
5- 6010 Pb only

Soil
260- BTE samples via 8020
327- 8015 Mod (TPH gas or diesel or 418.1)
152- 6010 for Pb, Cd, Cr
264- 8310 Naphthalene, Acenaphthene, Fluoranthene, and Benzo(a)pyrene
60- 8080 PCBs
7- 8010 tetrachloroethylene, tand 111-trichloroethane
10- 1020 flammability
3- HPLC (method supplied by client 2/16) for PGDN and 2-nitrodiphenylamine, Samples to arrive after March
40- 6010 Pb only

Protocol : SW-846 with NEESA Level C data package. See me if you need a copy of the NEESA Requirements. I have the June 88 version. Site specific QC required. Batch in SDGs.

QC : Yes! See handout submitted to;
JMB,MN,Bill,BarbM,MC,CD,MS,MIK,PJ,TM,BO

Turnaround : FAX 7 working days from sample receipt Hard Copy 30 calendar days from SDG closure EDD Not applicable

File: Y:OHM-HAWAII.PCC
Date: 01/24/96

Penalties : See PROJQC, page 2

CRDLs : Yes! See minimum detection limits in handout submitted to;
JMB,MN,Bill,BarbM,MC,CD,MS,MIK,PJ,TM,SO

Holding Time: Routine SW-846 from collection.

Deliverables: NEESA Level C

Comments :

All stock pile samples must be thoroughly homogenized prior to analysis.

UPDATES will be added (with the date) to this file.

Pam, see MarkS regarding special test codes for low level metals prep! Something like, LXWM1 for the digestion. Also, these samples will be batched in SDGs. See MarkS.

First tentative schedule starting 3/2 or 3/3:

90 soils - BTE (8020) + X
40 soils - TPH Gas
50 soils - TPH Diesel OR 418.1
50 soils - 8310 PAHs
40 soils - Pb only (6010)

3/15/95 sh;

Art Taddeo requested pricing for TCLP Pb, Cd, and Cr (to comply with TCLP regulations). I provided pricing for soils at $130.00 ea.

10/26/95 ds

Data packs are to be sent to:

Kim Osgood
OHM
20015 72nd Ave.S
Kent, WA 98032

Reports are to be faxed to Bob Rooks at 808-682-1880 in HI.

Appendix III

Example of SDG Paperwork

PRE-PACKAGE CHECKLIST

SDG# GTL27 open ☐ closed ☑

Work Order #(s)	Fax Due Date(s)
9603/2	1/16/96

Due to Office: 2/7/96

Due to client: 2/9/96

E.D.D.: yes ☐ Custom or CLP *(circle one)* no ☑

If yes, can the package ship without the disk? _____

Full Deliverables? yes ☑ no ☐

If no, what forms/raw data are required? _____

No. of Copies: 1

Special package requests:

Assigned Document controller: _____

THIS INFORMATION WILL BE USED FOR REPORTING/BILLING* (SEE BELOW)

NAME: Groundwater Technology GSE
ADDRESS: 19033 W Valley Hwy D-104
Kent WA 98302
ATTENTION: Diana Spence
PROJECT NAME: Bangor Site A
PROJECT CONTACT: Stan Thompson
TELEPHONE/FAX: 360 779 9564 / 360-779-1676
JOB/P.O. NO.: 83001-2153 110
SAMPLER (SIGNATURE): (PRINTED NAME) Daryl Braun

CHAIN OF CUSTODY RECORD: 1313
WORK ORDER ID: 83-01-2153 PAGE 1 OF 1
DATE 1-8-96

Laucks Testing Laboratories, Inc.
SUBMITTED AT: 4 [illegible] | [illegible]

TESTS TO PERFORM: M8330

LAB SAMPLE	SAMPLE ID / LOCATION	DATE	TIME		NO. OF CONTAINERS	OBSERVATIONS, COMMENTS, SPECIAL INSTRUCTIONS
1)	INF-CL-209-010896	1-8-96	0900	2L	2L	Fax Results to 360-779-1676

INSTRUCTIONS
1. USE ONE LINE PER SAMPLE.
2. BE SPECIFIC IN TEST REQUESTS.
3. CHECK OFF TESTS TO BE PERFORMED FOR EACH SAMPLE.

*BILLING INFORMATION, IF DIFFERENT THAN ABOVE
NAME
ADDRESS
ATTN:
CITY, STATE, ZIP

TURNAROUND REQUEST
- [ ] 24-48 HRS (100% SUR)
- [x] 5 DAYS (50% SUR)
- [ ] STD 10-11 DAYS
- [ ] OTHER

TOTAL NO. OF CONTAINERS: 2L
CHAIN OF CUSTODY SEALS? [ ] YES [ ] NO [ ] NA
SHIPPED VIA [ ] UPS [ ] FED EX [ ] BUS [ ] HAND [ ] _____
TEMPERATURE _____ [ ] AMBIENT [ ] REPRESENTATIVE

RELINQUISHED BY (SIGN AND PRINT)	DATE / TIME	RECEIVED BY (SIGN AND PRINT)	DATE / TIME
Daryl Braun	1-8-96 1600	Pony Express	
		PAM R. JOHNSON, Laucks Testing Labs	1/9/96 0936

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

?:\PM\Holly\docs\samplememo.doc

Initial once samples are checked in _____

DATE RECEIVED: 1/9/96
TIME RECEIVED: 0726
CLIENT NAME: GSI
SDG # GTI27
COC # N/A

SAMPLE LOG-IN DATE: 1/9/96
WORKORDER #: 9601313
CLIENT PROJECT: Danger Site A
AIRBILL ATTACHED?:(#) 2657 6393
RECEIVED BY: B

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

- ☐ (1) Not enough sample sent for proper analysis. #s affected: _____
- ☐ (2) Sample Bottle received broken and/or cap not intact. _____
- ☑ (3) Custody seal: Absent ____ Present/Intact ✓ Present/Broken _____
- ☐ (4) Any temperature out of compliance: _____
- ☐ (5) Sample received outside of holding time. _____
- ☐ (6) Sample not properly preserved. pH = ___. Wrong preservative used. _____
- ☐ (7) Illegible sample numbers or label missing from bottles. _____
- ☐ (8) Identification on bottle same as identification on paperwork: yes: _____ no: _____
- ☐ (9) Incomplete instructions received with sample(s), i.e., no Request for Analysis, no Chain-of-Custody. _____
- ☐ (10) Samples received in improper container. _____
- ☐ (11) Samples held in field before receipt by Lab. Days (specify) _____
- ☐ (12) Air Bubble(s) in ___ of ____ samples for volatiles analysis. _____
- ☐ (13) Other _____

CORRECTIVE ACTION: (Check applicable item(s))

		Correction action taken by: Initials	Date
☐	(1) Client informed verbally (Client Services).		
☐	(2) Client informed by memo/letter/fax (Client Services).		
☐	(3) Sample processed "as received" (Sample Entry).		
☐	(4) Re-sampling requested of client (Client Services).		
☐	(5) Samples placed "on hold" until further notice (Sample Entry/Client Services).		
☐	(6) NOTE IN NARRATIVE. See temperature/pH login sheet. (Sample Entry).		
☐	(7) Other (Specify) _____		

* When complete (within 24 hours of nonconformance) forward to QA. Original to be forwarded to initiator to be included in transmittal file.

Comments:

Laucks Testing Laboratories, Inc., Supplemental Sample Receipt Log

Work Order Number: 9601313
Assigned SDG Number: [illegible]

Temperature	pH or Bottle Types								
	8330								
1 2 0 1 2 3	2) 7								

Allowable temperature and pH ranges (neutral pH defined as a value between 5 and 9)

Temperature	Allowable temperature range is 4± 2 degrees Celsius
Acid Preserved pH	pH must be less than 2
Base Preserved pH	pH must be greater than 12

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-4201

Title: **Package Deliverables for all Reporting Levels**

Revision history:

Number	Date
000	1/31/96

Written by: _____ Date: 1-31-96
Tom Marino

Reviewed by: _____ Date: 1-31-96
Charlene Nix

Reviewed by: _____ Date: 1-31-96
Harry Romberg, QC Officer

Approved by: _____ Date: 1/31/96
Karen Kotz, Laboratory Director

UNCONTROLLED

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1. Introduction and Scope

1.1 This SOP descibes the contents of the different levels of reports at Laucks Testing Laboratories, Inc.

1.2 Complete CLP-type data package (level IV):

A complete data package (level IV) submitted to a client consists of a cover page, a narrative, chain-of-custody copies, an index, and a separate section for each analytical fraction containing all forms and raw data. The entire package is paginated sequentially beginning with #1.

1.3 Forms only data package (level III):

A "forms only" data package (level III) submitted to a client contains all of the above with the exception of the raw data.

1.4 Database Report (level II):

A database report (level II) contains forms generated from the database and includes many of the forms in a level III or IV package. The contents are indicated in Appendix I and are not described further.

1.5 Paper Job Report (level I):

A "paper job" report is created for special chemistry and food chemistry and usually contains a format specified by the client or results only. The contents are indicated in Appendix I and are not described further.

2. Description of Contents of Level III and IV packages

Detailed below are the elements that may be included in a level III or level IV package. Appendix I contains tables that signify whether an element is incorporated into a final report. Client specific requests may dictate that some elements may be added or deleted and these are documented during project initiation in the LIMs system.

2.1 Cover Page

The package cover page will contain the laboratory name, client name, work order numbers, SDG number and the date.

2.2 Narrative

2.2.1 Sample receipt and Identification:

This section lists all client sample names, the corresponding laboratory sample names and the analyses requested for each sample. (The analyses are generally abbreviated to three letters.)

2.2.2 Analytical request key:

This section defines the abbreviations listed in the above section.

2.2.3 Sample Identification on Forms:

This sections is used to explain any abbreviations to client sample names on any of the forms (occasionally forms software does not accommodate lengthy client sample I.D.s).

2.2.4 General remarks on organic analysis:

These are stock comments contained in the narrative template describing general analysis conditions for each of the requested organic fractions.

2.2.5 Specific remarks on organic analysis:

These are comments written for each organic analytical fraction describing any anomalies, deviations from the specified method, dillutions, holding time violations, corrective actions etc. These comments are written by the respective analysts.

2.2.6 General remarks on inorganic analysis

These are stock comments contained in the narrative template describing general analysis conditions for each of the requested inorganics fractions.

2.2.7 Specific Remarks on inorganic analysis:

These are comments written for each inorganic analytical fraction describing any anomalies,deviations from the specified method, dilution's, holding time violations,corrective actions etc. These comments are written by the respective analysts.

2.2.8 Release of data:

This page is signed by both the respective project manager and the technical director. also contained on this page is information on who to contact regarding specific questions as well as the laboratory telephone and fax numbers

2.3 Chain-of-Custody Copies:

This section contains the chains of custody received with the samples as well the laboratory receipt and temperature logs.

2.4 Index:
The index should list all data fractions and sub-fractions with the corresponding page numbers.

2.5 Organic fractions

Level IV Organic data packages are subdivided into five sections: QC Summary, Sample Data, Standards Data, Raw QC Data, and Bench Sheets.

Level III (forms only) organic data packages contain only the **forms** from these sections. No bench sheets or raw data are provided.

Only Volatiles, Semi-volatiles and Pesticides/PCBs have official CLP form numbers and protocol. Every effort is made to ensure that the same information appears on forms for all other fractions.

NOTE: the form numbers that appear below are seen only on forms for Volatiles, Semi-Volatiles and Pesticides/PCBs. Forms for all other fractions contain the same information but no actual form numbers.

2.5.1 QC Summary:

The QC Summary contains the following forms:

a.) Form II: Surrogate recovery report
b.) Form III: MS/MSD Recovery report
c.) Blank spike report
d.) Form IV: Method blank summary
e.) Form V: Tuning and Mass Calibration Standard
f.) Form VIII: Internal standards Area Summary

2.5.2 Sample Data:

Sample data contains the following forms and data

a.) Form I (analysis data sheet) including TICs
b.) Raw Data

2.5.3 Standards Data:

The standards data below are divided into two formats: Volatile/Semi-Volatile and Pesticide/PCB. Every effort is made to ensure that forms for all other fractions adhere closely to whichever of the two formats is most applicable.

2.5.3.1 Volatile/Semi-Volatile

a.) Form VI and Initial Calibration Data
b.) Form VII and Continuing Calibration Data

2.5.3.2 Pesticide/PCB

a.) Form VIII: Pesticide Analytical Sequence
b.) Form IX: Pesticide/PCB Standards Summary
c.) Form X: Pesticide/PCB Identification (positive results)
d.) Pesticide standard chromatograms and data system printouts for Evaluation of standard mix A,B, and C
e.) Pesticide standard chromatograms and data system printouts for individual standard mix A, and B
f.) Pesticide Standard Chromatograms and data system printouts for all multi-response pesticides/PCBs and quantitation standards
g.) A copy of the computer reproduction or strip chart recorder output covering the 100 fold range

2.5.4 Raw QC Data:

The Raw QC Data below are divided into three formats: Volatile, Semi-Volatile, and all other fractions.

2.5.4.1 Volatile
- a.) BFB
  - 1.) Bar graph spectrum
  - 2.) Mass listing
  - 3.) RIC: Reconstructed Total Ion Chromatogram
- b.) Blank Data
  - 1.) Form I including TICs
  - 2.) Raw data
- c.) Matrix Spike Data
  - 1.) Form I
  - 2.) Raw data
- d.) Matrix Spike Duplicate Data
  - 1.) Form I
  - 2.) Raw data

2.5.4.2 Semi-Volatile
- a.) DFTPP
  - 1.) Bar graph spectrum
  - 2.) Mass listing
  - 3.) RIC: Reconstructed Total Ion Chromatogram
- b.) Blank Data
  - 1.) Form I including TICs
  - 2.) Raw data
- c.) Matrix Spike Data
  - 1.) Form I
  - 2.) Raw data
- d.) Matrix Spike Duplicate Data
  - 1.) Form I
  - 2.) Raw data

2.5.4.3 All other fractions
- a.) Blank Data
  - 1.) Form I
  - 2.) Raw data

b.) Matrix Spike Data
  1.) Form I
  2.) Raw data
c.) Matrix Spike Duplicate Data
  1.) Form I
  2.) Raw data

2.5.5 Bench Sheets:

The bench sheets section contains any miscellaneous paper work such as log book pages, in-house tracking sheets, in-house chains of custody, extraction bench sheets etc.

2.6 Inorganics Fractions:

There are two kinds of inorganics fractions: Metals and Miscellaneous Inorganics (conventionals).

2.6.1 Metals

2.6.1.1 Cover Page:

This page lists all samples analyzed and is signed and dated by the analyst

2.6.1.2 Inorganics Analysis Data Sheet
a.) forms I

2.6.1.3 Quality Control Data
a.) Form II (part 1): Initial and Continuing Calibration Verification
b.) Form II (part 2): CRDL Standard for AA and ICP
c.) Form III: blanks
d.) Form IV: ICP Interference Check Sample
e.) Form V (part 1): Spike Sample Recovery
f.) Form V (part 2): Post Digest Spike Sample Recovery
g.) Form VI: Duplicates
h.) Form VII: Laboratory Control Sample
i.) Form VIII: Standard Addition Results
j.) Form IX: ICP Serial Dilutions
k.) Form XIII: Preparation Log
l.) Form XIV: Analysis run Log

2.6.1.4 Quarterly Verification of Instrument Parameters
- a.) Form X: Instrument Detection Limits (quarterly)
- b.) Form XI (part 1): ICP Inter element Correction Factors (annually)
- c.) Form XI (part 2): ICP Inter element Correction Factors (annually)
- d.) Form XII: ICP Linear Ranges (quarterly)

2.6.1.5 Raw Data
- a.) ICP
- b.) Graphite Furnace
- c.) Mercury
- d.) Cyanide

2.6.1.6 Digestion and Distillation Logs
- a.) ICP
- b.) Graphite Furnace
- c.) Mercury
- d.) Cyanide

2.6.2 Miscellaneous Inorganics

2.6.2.1 Cover Page

This page lists all samples analyzed and is signed and dated by the analyst.

2.6.2.2 Inorganics Analysis Data Sheet

2.6.2.3 Quality Control Data
- a.) Preparation blanks database Report
- b.) MS/MSD Report
- c.) MS/Duplicate Database
- d.) SRM Report

2.6.2.4 Raw Data

The Miscellaneous Inorganics raw data is divided into sections by analytes.

Appendix I

Laucks Testing Laboratories
Organics
Levels of Reporting

	Level I	Level II	Level III	Level IV = CLP
Narrative			Y	
Chain-of-Custody	Y	Y	Y	Y
Method Reference	Y	Y	Y	Y
Results	Y	Y	Y	Y
Surrogate Rec.		Y	Y	Y
Method Blank		Y	Y	Y
Blank Spike		Y	Y	Y
MS/MSD		Y	Y	Y
Tune			Y	Y
Initial Calib.			Y	Y
Cont. Calib.			Y	Y
IS Area				Y
Raw Data			Optional	Y
Chromatograms			Optional	Y
Bench Sheets				Y

Y indicates that this element is present in the hardcopy delivered to the client.

Laucks Testing Laboratories
Metals
Levels of Reporting

	Level I	Level II	Level III	Level IV = CLP
Narrative			Y	Y
Chain-of-Custody	Y	Y	Y	Y
Method Reference	Y	Y	Y	Y
Results	Y	Y	Y	Y
Method Blank		Y	Y	Y
SRM/LCS/BS		Y	Y	Y
MS/Dupe		Y		Y
MS/MSD			Y	Y
ICV			Y	Y
ICB			Y	Y
CCV			Y	Y
CCB			Y	Y
CRA			Y	Y
CRI			Y	Y
Post Spike			Y	Y
GFAA MSA			Y	Y
ICP Ser. Dil'n.			Y	Y
IDLs			Y	Y
Interelement Corr. Factors				Y
Linear Range				Y
Prep Log				Y
Run Log				Y
Raw Data				Y
Bench Sheets				Y

Y indicates that this element is present in the hardcopy delivered to the client.

Laucks Testing Laboratories
Wet Chemistry
Levels of Reporting

	Level I	Level II	Level III	Level IV = CLP
Narrative			Y	Y
Chain-of-Custody	Y	Y	Y	Y
Method Reference	Y	Y	Y	Y
Results	Y	Y	Y	Y
Method Blank		Y	Y	Y
SRM/LCS/BS		Y	Y	Y
MS/Dupe		Y		Y
MS/MSD			Y	
ICV			Y	Y
ICB			Y	Y
CCV			Y	Y
CCB			Y	Y
Raw Data				Y
Bench Sheets				Y

Y indicates that this element is present in the hardcopy delivered to the client.

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-6006

Title: Cation-Exchange Capacity of Soils (Sodium Acetate) by SW 846 Method 9081

Revision history:

Number	Date
0	1/25/99

UNCONTROLLED

Written by: Harry Romberg Date: 1-25-99
Harry Romberg, Quality Assurance Officer

Reviewed by: Raul N Date: 1/25/99
Raul Nino, Analyst

Approved by: Kathy Kreps Date: 1/25/99
Kathy Kreps, Laboratory Director

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QC SUMMARY TABLE 8

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FLOW CHART 10

1. Introduction and Scope

1.1 Scope

1.1.1 LTL-6006 (SW 846 Method 9081) is applicable to most soils, including calcareous and noncalcareous soils. This method only applies to the Cation Exchange Capacity extraction. The actual analysis method should be referred to for quantitation of sodium in the extract.

1.2 Method Description

1.2.1 The soil sample is mixed with an excess of sodium acetate solution, resulting in an exchange of the added sodium cations for the matrix cations. Subsequently, the sample is washed with isopropyl alcohol. An ammonium acetate solution is then added, which replaces the adsorbed sodium with ammonium. The concentration of displaced sodium is then determined by flame atomic absorption spectroscopy or ICP.

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

1.3 Sample Collection, Sample Storage, Holding Times

1.3.1 No holding times or preservation are specified for this method.

1.4 Definition of Terms

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

1.4.2 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. This is followed by the date, analysis, matrix and sequence number (i.e. BmmddyyCECS01).

1.4.3 CLP - Contract Laboratory Program - The USEPA program that contracts with laboratories to provide laboratory services. The term has come to mean a much broader set of methods and deliverables. In context of this SOP, CLP means procedures or operations which are detailed in the CLP contract and which are extended to a broader working definition.

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

1.4.5 RPD - Relative percent difference, in this case between duplicate analyses, calculated as the difference between replicate sample determinations divided by the average calculated as a percent.

2. Equipment List and Standards

2.1 Equipment

- 50 mL centrifuge tubes and stoppers
- mechanical shaker
- 100 mL class A volumetric flasks

2.2 Reagents

2.2.1 Sodium acetate (NaOAc), 1.0 N: Dissolve 136 g of $NaC_2H_2O_2 \cdot 3H_2O$ (or 82 g anhydrous $NaC_2H_2O_2$) in water and dilute it to 1,000 mL. The pH of this solution should be 8.2. If needed, add a few drops of acetic acid or NaOH solution to bring the reaction of the solution to pH 8.2.

2.2.2 Ammonium acetate (NH_4OAc), 1 N: Dilute 114 mL of glacial acetic acid (99.5%) with water to a volume of approximately 1 liter. Then add 138 mL of concentrated ammonium hydroxide (NH_4OH) and add water to obtain a volume of about 1,980 mL. Check the pH of the resulting solution, add more NH_4OH, as needed, to obtain a pH of 7, and dilute the solution to a volume of 2 liters with water.

2.2.3 Isopropyl alcohol: 99%.

3. Safety precautions and Waste Disposal

3.1 Safety Precautions

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

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

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

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

3.2 Waste Disposal

3.2.1 Isopropyl alcohol solutions should be disposed of in the solvent waste container.

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

4. Calibration and Quality Control

4.1 Method Blanks

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

4.1.2 Corrective action

4.1.2.1 Corrective action may necessitate re-preparation and re-analysis of the sample set. For example if an analyte were found in the blank but not in any of the associated samples then sample group may not require re-analysis. In addition, if sample levels exceed 20 times the blank, the level of contamination may be considered insignificant. In any case, if re-preparation and re-analysis is not being undertaken, the analyst must first discuss the issue with the Quality Control Officer. It is the laboratory's responsibility to ensure that method interferences caused by contaminants in acids, solvents, reagents, glassware, and other sample processing hardware leading to discrete artifacts and/or elevated baselines in the analytical run be minimized. In the extreme case of chronic contamination, blanks may have to be analyzed from each stage of the sample processing to determine the contamination source so it can be eliminated. In all cases where blank contamination exceeds the control limit, a narrative comment must be made which documents the corrective actions taken.

4.2 Sample Duplicate

4.2.1 Criteria

4.2.1.1 At least one duplicate sample per 10 samples is required. RPD values are calculated in a manner similar to MS/MSD RPDs:

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

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

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

4.2.2 Corrective action

4.2.2.1 If a trend in out of control RPD values is observed, the methods used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable reproducibility. Generally, if recoveries are in control and no analyte of interest was detected in any of the samples, no immediate action will be taken on that sample set. If integrity of reported sample values is in doubt, re-analysis may be called for. Corrective actions should be discussed with the Quality Control Officer.

5. Operation procedures

5.1 Extraction Procedure

5.1.1 Weigh 4 g of medium or fine-textured soil or 6 g of coarse-textured soil and transfer the sample to a 50-mL, round-bottom, narrow-neck centrifuge tube. (A fine soil has >50% of the particles <0.074 mm, medium soil has >50% >0.425 mm, while a coarse soil has more than 50% of its particles >2 mm.

5.1.2 Add 33 mL of 1.0 N NaOAc solution, stopper the tube, shake it in a mechanical shaker for 5 min, and centrifuge it until the supernatant liquid is clear.

5.1.3 Decant the liquid, and repeat Paragraph 5.1.2 three more times.

5.1.4 Add 33 mL of 99% isopropyl alcohol, stopper the tube, shake it in a mechanical shaker for 5 min, and centrifuge it until the supernatant liquid is clear.

5.1.5 Repeat the procedure described in Paragraph 5.1.4 two more times.

5.1.6 Add 33 mL of NH_4OAc solution, stopper the tube, shake it in a mechanical shaker for 5 min, and centrifuge it until the supernatant liquid is clear. Decant the washing into a 100-mL volumetric flask.

5.1.7 Repeat the procedure described in Paragraph 5.1.6 two more times.

5.1.8 Dilute the combined washing to the 100-mL mark with ammonium acetate solution and determine the sodium concentration by atomic absorption, emission spectroscopy, or an equivalent method.

5.2 Instrumental Analysis

5.2.1 Specifics of the instrumental analysis are contained in the appropriate SOP.

6. Reports

6.1 Data Packet Organization

6.1.1 Sample reports vary depending upon the needs of the client. They may be as simple as a standard laboratory report with only a QC summary (generated from QC_DB) or they may be more thorough and include instrument calibration information and raw data if a full data package is required.

6.2 Quality Control Reports

6.2.1 All results for quality control tests are entered into the lab data base using the QC_DB program. Printouts of all data entered must be included in the data packet. The routine minimum for this analysis is a method blank report and a duplicate report.

6.3 Sample Result Reports

6.3.1 Data Qualifying Flags

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

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

Appendix I

QC Summary Table

Laucks Testing Laboratories

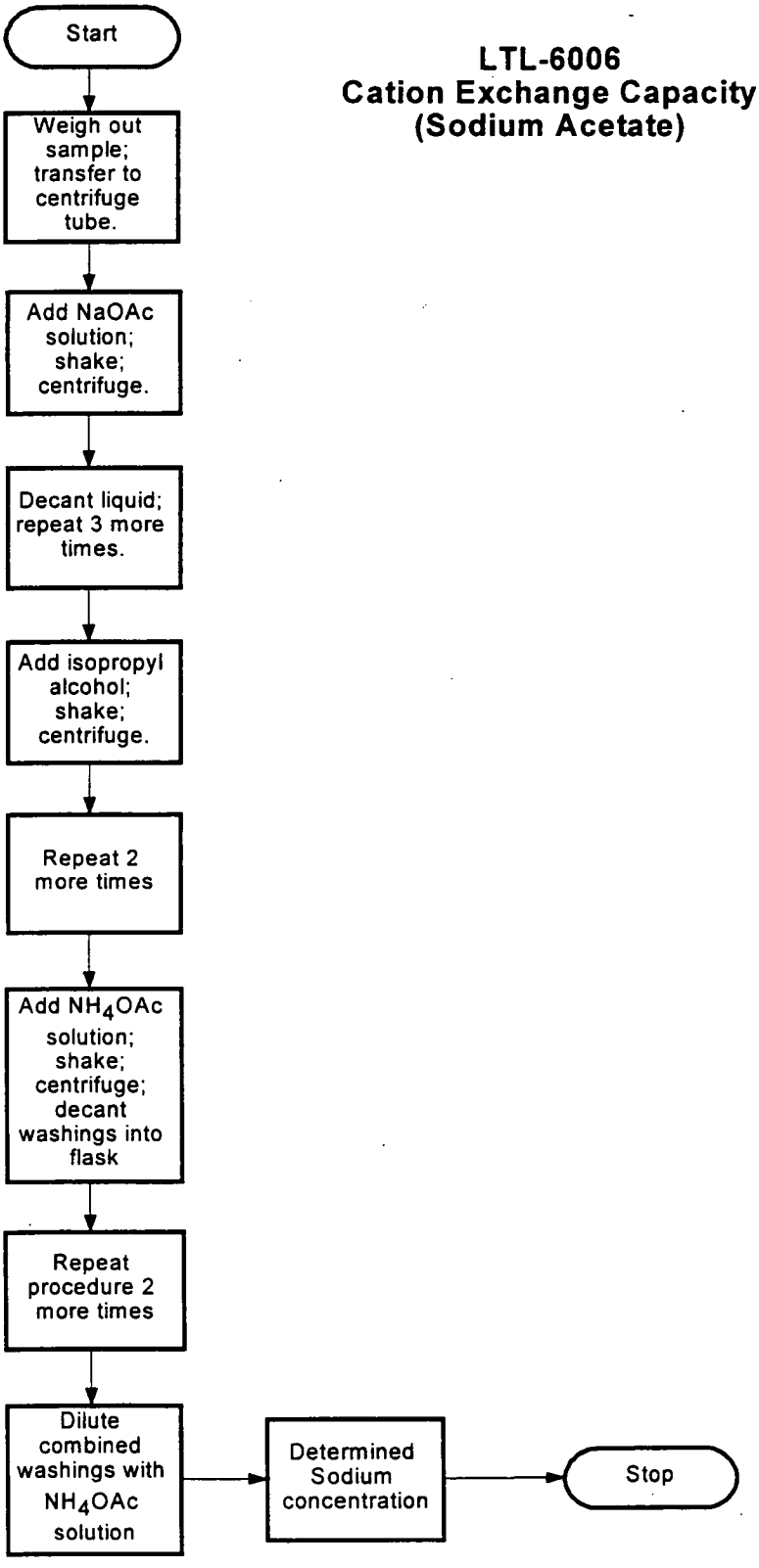
Method LTL-6006 QA Requirements and Corrective Actions

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Method Blank	None	No blank above reporting limit	1 per 20 samples or one per batch	reanalyze unless samples >20X blank	QC_DB and sample report
Duplicate % Difference or RPD	None	See QC_DB database	1 per 10 samples or one per batch	Discuss with QC Officer	QC_DB and sample report

Appendix II

Flow Chart

LTL-6006
Cation Exchange Capacity
(Sodium Acetate)



LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-7003

Title: **Inorganic Glass Cleaning Procedure**

Revision history:

Number	Date
2	04/09/97
1	01/05/88

Written by: Bill Lundberg, Inorganics Supervisor Date: 4/9/97

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

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

UNCONTROLLED

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1. Introduction and Scope

1.1 Method Description

1.1.1 The purpose of this SOP is to define the procedures used in the inorganics department for the cleaning of glassware. The objective is to define a specific method of cleaning that is adapted to both the substances that are to be removed, and the determination to be performed.

1.2 Definitions

1.2.1 DIW = deionized water

2. Equipment List and Standards

2.1 Standard laboratory glassware, including but not limited to:

2.1.1 Volumetric flasks
Beakers
Funnels
Separatory funnels
Kjeldahl flasks
Nessler tubes
Erlenmeyer flasks
Burets
BOD bottles
Distillation Apparatus

3. Safety precautions and Waste Disposal

3.1 Safety Precautions

3.1.1 Several cleaning procedures or soaking procedures require the use of a chromic acid cleaning solution, concentrated HNO_3 or H_2SO_4. USE APPROPRIATE SAFETY PRECAUTIONS FOR ACID USE! Wear safety glasses, lab coat, and gloves.

3.1.2 Some oily samples or profile samples may require the use of acetone or chloroform to clean the glassware. AGAIN, USE APPROPRIATE SAFETY PRECAUTIONS FOR SOLVENT USE! Wear safety glasses, lab coat, and gloves. Dispose of solvent waste in appropriate solvent waste barrel.

3.1.3 Some profile-type samples may not clean up even with solvent washes, and therefore may need to be baked in the muffle furnace at 500 degrees C for 1-3 hours.

3.1.4 CAUTION: Be sure to evaporate any residual solvent from glassware before putting in muffle furnace.

3.1.5 Do not put soft glass (non-pyrex, kimax, etc.) in a muffle furnace or it will shatter. Take appropriate precautions with hot materials.

4. Operation procedures

4.1.1 All glassware must be scrupulously cleaned. The analyst that performs each specific analysis is responsible for the proper cleaning of his or her own glassware. Glassware used in routine analysis is kept separate from the general use glassware. Specific cleaning procedures are listed by type of analysis.

4.1.2 ALKALINITY

4.1.2.1 Glassware Erlenmeyer flasks

4.1.2.2 Buret

4.1.2.3 Cleaning Procedures - Rinse with DIW.

4.1.2.4 Air dry.

4.1.3 AUTO ANALYZER (refer to the applicable analytical SOP)

4.1.4 BOD

4.1.4.1 Glassware BOD bottles

4.1.4.2 Glass pipettes

4.1.4.3 Cleaning procedure - Wash with hot tap water and Alconox.

4.1.4.4 Rinse with hot tap water.

4.1.4.5 Rinse with DIW.

4.1.4.6 Air dry.

4.1.5 COD

4.1.5.1 Procedure for Glassware Condensers, Erlenmeyer flasks, Buret

4.1.5.2 Cleaning Procedure - Rinse well with DIW only.

4.1.5.3 Soaking Procedure - Acid soak flasks with C.O.D. acid for 10 minutes prior to use. Rinse well with DIW.

4.1.6 CYANIDE

4.1.6.1 Procedure for Glassware, Volumetric flasks, Distillation apparatus

4.1.6.2 Cleaning procedure for Flasks - DIW rinse only.

4.1.6.3 Distillation apparatus - occasional H_2SO_4 and DIW wash, but generally DIW rinsed only.

4.1.7 Hexavalent Chromium

4.1.7.1 Cleaning procedure - Wash with hot tap water and Alconox.

4.1.7.2 Rinse well with DIW.

4.1.7.3 Air dry.

4.1.8 FORMALDEHYDES

4.1.8.1 Procedure for GlasswareBeakers, Erlenmeyer flasks, Volumetric flasks, Test tubes

4.1.8.2 Cleaning procedures - ,Rinse well with DIW only.

4.1.8.3 Never contaminate glassware with HNO_3 .

4.1.9 HARDNESS

4.1.9.1 Procedure for Glassware, Erlenmeyer flasks, Buret

4.1.9.2 Cleaning procedure - Rinse with DIW only.

4.1.10 KJELDAHL NITROGEN-LOW LEVELS (TKN AND AMMONIA)

4.1.10.1 Procedure for Glassware, Kjeldahl distillation apparatus, Kjeldahl flasks, Erlenmeyer, flasks or beakers

4.1.10.2 Cleaning procedure for Kjeldahl flasks - pre-distill with DIW and NaOH, and do final DIW rinses.

4.1.10.3 Beakers or Erlenmeyer flasks- DIW rinse only.

4.1.11 OIL AND GREASE

4.1.11.1 Procedure for Glassware, Volumetric flasks, Beakers, Funnels, Separatory funnels, Soxhlet digestion apparatus

4.1.11.2 Cleaning procedure for Vol. flasks, funnels, soxhlet apparatus- freon rinse 3-4 times.

4.1.11.3 Cleaning procedure for Beakers, separatory funnels- hot tap water and Alconox, rinse well with hot tap water, and dry.

4.1.12 METALS (INCLUDING HYDRIDES AND MERCURY)

4.1.12.1 Procedure for Glassware, Volumetric flasks, Beakers, Erlenmeyer flasks, BOD bottles, Digestion caps or watchglasses

4.1.12.2 Cleaning procedure - Rinse well with DIW only.

4.1.12.3 OPTIONAL:

4.1.12.4 If oily or difficult to clean, wash with acetone, scrub with hot tap water and Alconox, and rinse well with DIW.

4.1.12.5 Concentrated acid wash and DIW rinsed.

4.1.12.6 Bake in muffle furnace at 550 degrees C for 1 hour and rinse well with DIW.

4.1.12.7 NOTE: Some metals glassware should not be cleaned with Alconox at all.

4.1.12.8 Soaking procedure - Acid soak all metals glassware in HNO_3 and DIW.

4.1.12.9 Fill glassware with DIW and add 4-8 mls HNO_3. Cover and store until next use.

4.1.13 PHENOL

4.1.13.1 Procedure for Glassware, Kjeldahl flasks, Erlenmeyer flasks

4.1.13.2 Cleaning procedure for Kjeldahl flasks - pre-distill with H_2SO_4 and catalyst, DIW rinse.

4.1.13.3 Cleaning procedure for Erlenmeyer flasks - acid H_2SO_4 wash and DIW rinse.

4.1.13.4 NOTE: Use only those Kjeldahl flasks designated for use in phenol distillations.

4.1.14 PHOSPHATE

4.1.14.1 Cleaning procedure for GlasswareBeakers

4.1.14.2 Acid wash with H_2SO_4 .

4.1.14.3 Rinse with DIW.

4.1.15 TOC (SOILS)

4.1.15.1 Glassware, TOC combustion boats

4.1.15.2 Cleaning procedure - Brush out last sample.

4.1.15.3 Incinerate boat in muffle furnace at 950 degrees C.

4.1.16 TOX

4.1.16.1 Cleaning procedure for Glassware, Volumetric flasks, Misc. TOX glass parts

4.1.16.2 Cleaning procedure - Soak in chromic acid solution.

4.1.16.3 Wash with hot tap water and alconox.

4.1.16.4 Rinse well with DIW.

4.1.16.5 Bake in muffle furnace at 400 degrees C.

4.1.16.6 Cool.

4.1.16.7 Store in glass teflon sealed container inside a dessicator.

5. References

5.1.1 See the applicable analytical or preparation SOP for specific cleaning issues and references.

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-7009

Title: **Microwave Assisted Acid Digestion of Aqueous Samples and Extracts Using SW846 Method 3015**

Revision history:

Number	Date
1.	04/20/95
2.	06/04/96
3	06/29/00

Revised by: Stan Dittrick, Chemist Date: 7/28/00

Reviewed by: Paul Swift, Inorganics Manager Date: 07/28/2000

Reviewed by: Harry Romberg, QC Officer Date: 7-28-2000

Approved by: Kathy Kreps, Laboratory Director Date: 07-28-2000

Controlled Document

No. 20 Assigned to: Tetra

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1. Introduction and Scope

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

1.2 This digestion procedure is used for the preparation of aqueous samples, mobility-procedure extracts, and wastes that contain suspended solids for analysis, graphite furnace absorption spectroscopy (GFAA), inductively coupled argon plasma spectroscopy (ICP), or inductively coupled argon plasma mass spectrometry (ICP-MS). The procedure is a hot acid leach for determination of available metals.

1.3 Samples prepared by Method 3015 using nitric acid digestion may be analyzed by GFAA, ICP or ICP-MS for the following:

Aluminum	Calcium	Lead	Selenium
Antimony	Chromium	Magnesium	Silver
Arsenic	Cobalt	Manganese	Sodium
Barium	Copper	Molybdenum	Thallium
Beryllium	Iron	Nickel	Vanadium
Cadmium	Zinc	Potassium	

1.4 Microwave power settings and times used in this SOP are based on manufacturers recommendation. Laucks system is different from the design used in both the SW846 and EPA CLP publications, in that it uses a 12 place carousel rather than 5.

2. Summary of Method

2.1 Nitric acid is added to an aqueous sample in a 120 mL Teflon® digestion vessel. The vessel is capped and heated in a microwave unit. After cooling, the vessel contents are filtered, or allowed to settle in a clean sample bottle for analysis.

3. Safety Procedures

3.1 These procedures involve the use of strong and/or hot acid solutions. The analyst must wear eye protection, a lab coat, and gloves to protect against burns.

3.2 Samples and spiking solutions may contain high levels of toxic metals and other unknown constituents. The analyst must take every precaution to avoid contact with these potentially hazardous materials and should wash hands thoroughly before eating or leaving the laboratory.

3.3 Very reactive or volatile materials may create high pressures when heated, causing the vessels to vent with potential loss of sample and analytes. Samples that contain carbonates or other carbon dioxide generating compounds may generate enough pressure to vent the vessel. If this situation is anticipated the analyst may wish to use a smaller sample or an alternative digestion method.

4. Equipment

- MDS-2100 Microwave Digestion system, CEM
- Capping station
- Carousel
- 120 mL Teflon® vessels and caps
- 50 or 100 mL graduated cylinder, acid washed
- Glass funnels, acid-washed
- Filter paper, Whatman No. 41 or equivalent
- Sample digestate bottles, acid washed or precleaned
- Analytical balance, 300 g capacity, minimum ± 0.01 g.

Note: All glassware used for this digestion must be prepared by the method stated in Inorganics Glassware Cleaning Procedures, LTL-7003.

4.1 All Teflon® vessels and caps are to be thoroughly rinsed with deionized water prior to digestions and shaken to remove any significant droplets in the vessels.

4.2 The microwave is to be calibrated by using the method stated in SW846 method 3051.

5. Reagents

5.1 Reagent Water - Reagent water will be interference free. All references to water in the method refer to reagent water unless otherwise specified.

5.2 Nitric acid (concentrated), HNO_3, Trace ICP analyzed

The method blank, prepared along with the samples, is used as a contamination check. Since the holding time for all analytes associated with this method have a holding time of 6 months, repreparation would not be an issue if contamination was traced to a specific reagent.

6. Microwave Sample Digestion Procedure

See Appendix A for sample digestion log.
See Appendix B for flowchart.
See Appendix C for QC Solutions

6.1 A 45 mL aliquot of well shaken sample is measured into a graduated cylinder. This aliquot is poured into the Teflon® digestion vessel with the number of the vessel recorded on the preparation sheet.

6.2 A blank sample of reagent water is treated in the same manner along with spikes , duplicates, and laboratory control samples. If only one graduated cylinder is used for all samples, the preparation blank is to be the last sample dispensed to assure the absence of sample carry-over.

6.3 Add 5 mL of concentrated nitric acid to each vessel and cap hand tight. For each microwave digestion run, a double ported lid is to accompany the sample that seems to have the most carbon. Weigh each capped vessel to the nearest 0.01 g.

6.4 Place the carousel in the microwave. Flush the pressure control line with water to remove any air bubbles. Attach the pressure control line, and the temperature probe to the digestion vessel with the double ported lid. Set the microwave on program 2 sw3015 (temperature). This first stage of the program increases the temperature from about 20°C to 160°C in ten minutes. In the next ten minutes the microwave heats the sample from 160°C to 170°C. Leave the samples in the microwave until it stops, to cool the samples.

6.5 Re-weigh the samples to determine the extent of sample loss, if any. If the weight of the bomb with the sample has decreased by more than 2.5g, discard and prepare the sample again.

6.6 Uncap the vessels and transfer the samples to an acid-cleaned or precleaned polyethylene bottle. If the digested sample contains particulate which may clog nebulizers or interfere with injection of the sample into an instrument, filter the sample or allow it to settle.

6.7 Record the start and finish time on the digestion log. Take the prepared samples to the metals analysis department. Record, the time and date when the digestates are relinquished, as well as the initials of the analyst accepting the digestates. Maintain a strict chain of custody on the digest log.

7. Quality Control

7.1 In each batch or SDG consisting of no more than 20 samples, a preparation blank is created. The blank should be labeled on the digestate bottle in the following way: B, date, MS (ICP-MS), ICP and the sequence number of the digestion. Example B022095MSW01.

7.2 Spiked samples should be employed to determine accuracy. In each batch or SDG consisting of no more than 20 samples, either matrix spike / sample duplicate (MS/Dup) or matrix spike /matrix spike duplicate (MS/MSD) samples will be prepared. For SW3015-specified quality control measures, MS/Dup samples will be prepared in the following way. Dispense a sample in triplicate. Two will be analyzed exactly as any other sample. The third must be spiked with the appropriate spike solutions, determined by the analyst. All glassware and digestate bottles must be marked appropriately. If MS/MSD samples are required instead of the SW846-specified MS/Dup, both the second and third aliquot must be spiked.

7.3 In each batch or SDG consisting of no more than 20 samples, prepare a Lab Control Sample (LCSW) in exactly the same manner as a sample. Each spiking solution is given its own unique number according to the page and line of the standards logbook which it is entered into. This number and the volume dispensed must be clearly recorded on the digestion log. A sample page from the standards logbook is included in Appendix C.

8. References

8.1 USEPA, Test Methods for Evaluating Solid Waste Physical/Chemical Methods, USEPA SW846, most recent version, Method 3015.

Appendix A, Sample LTL-7009 Digestion Log

Testing Laboratories, INC.	DATE TIME	DATE TIME	METHOD	ANALYST	ANALYSIS
SDG #:			SW846 3015		Microwave Digestion
MATRIX: WATER					

Bomb #	Sample ID	Samp Vol (mL)	Final Vol (mL)	Color		Clarity		Bomb & Sample (g)	Final Bomb (g)	Δ (g)	Δ >2.5 g ?	Bottle #
				Int	Fin	Int	Fin					

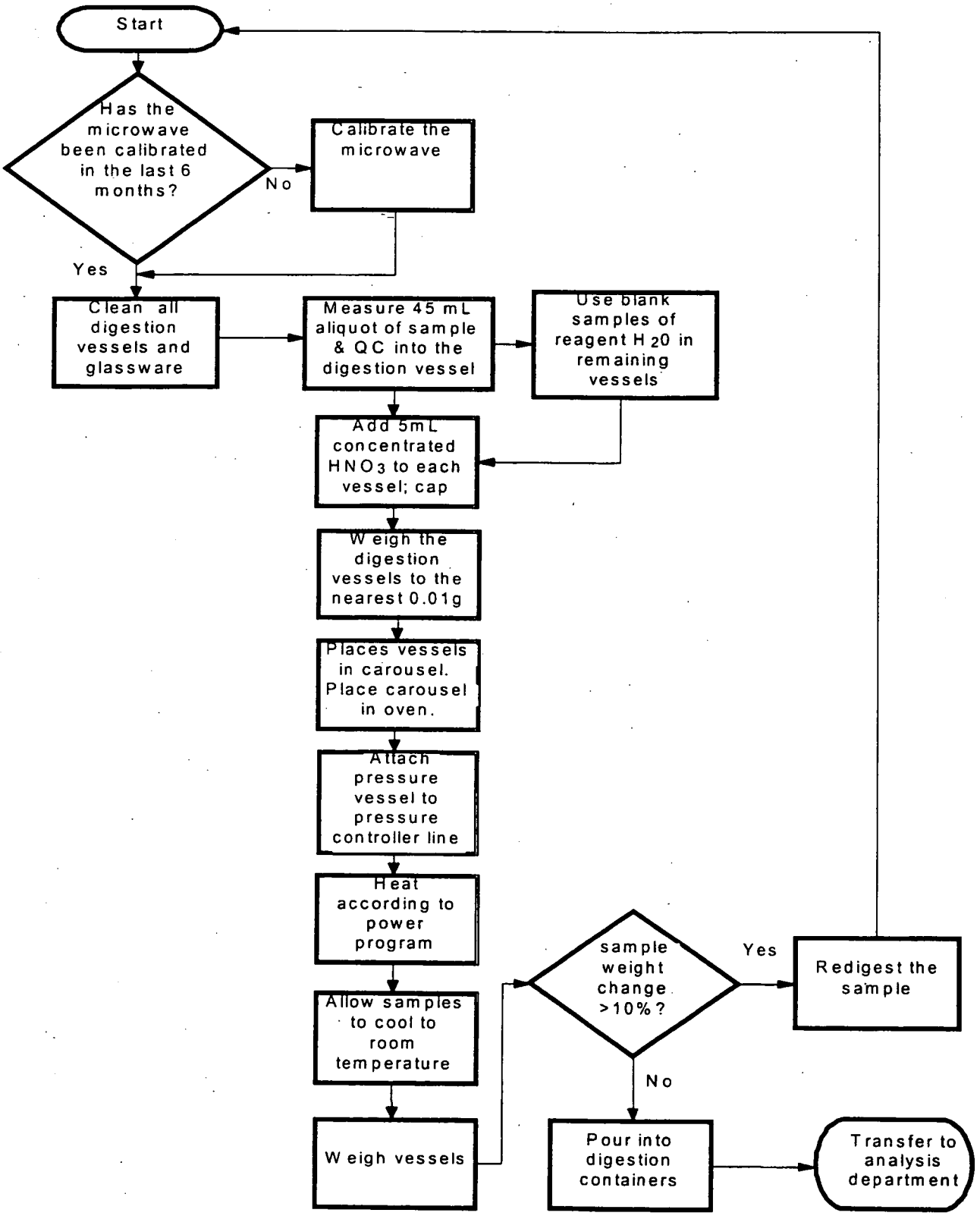
Chain of Custody	Digest Relinquished Date / Time : Analyst	Digest Accepted :Analyst	Digest Disposed :Date
Reagents Lot Number	HNO_3	HCl	H_2O_2

COLOR: 1 = Red, 2 = Blue, 3 = Yellow, 4 = Green, 5 = Orange, 6 = Violet, 7 = White, 8 = Brown, 9 = Gray, 10 = Black, 11 = Colorless.

CLARITY: 1 = Clear, 2 = Cloudy, 3 = Opaque.

P:\metals\bnchshee\sw3015.doc

Appendix B, Flowchart for LTL-7009


Start
Has the microwave been calibrated in the last 6 months?
No
Calibrate the microwave
Yes
Clean all digestion vessels and glassware
Measure 45 mL aliquot of sample & QC into the digestion vessel
Use blank samples of reagent H20 in remaining vessels
Add 5mL concentrated HNO3 to each vessel; cap
Weigh the digestion vessels to the nearest 0.01g
Places vessels in carousel. Place carousel in oven.
Attach pressure vessel to pressure controller line
Heat according to power program
Allow samples to cool to room temperature
Weigh vessels
sample weight change >10%?
Yes
Redigest the sample
No
Pour into digestion containers
Transfer to analysis department

Appendix C, Quality Control Solutions

ICP Spikes

- The CLP spike solution for ICP digest was made by diluting 10 mLs of CLPP-SPK-1, 10 mLs of CLPP-SPK-2, and 10 mLs of CLPP-SPK-3 to 100 mLs with Type II water.

- Add 1000 µLs of CLP spike solution to water samples and 2000 µLs of CLP spike solution to soil samples prior to digestion.

ICP Analytes

Analyte	Concentration in Digest (ppb)	Analyte	Concentration in Digest (ppb)
Ag	50	Fe	1000
Al	2000	Mn	500
As	2000	Ni	500
Ba	2000	Pb	500
Be	50	Se	2000
Cd	50	Sb	500
Co	500	Tl	2000
Cr	200	V	500
Cu	250	Zn	500

Table based on a final volume of 100 mL for water digests, and 200 mL for soil digests.

GRAPHITE FURNACE SPIKES

- Add the analytes listed in the table below to a 100 mL volumetric flask which contains ~50 mL of Type II water and 5 mL of HNO_3. Dilute to volume with Type II water.

Analyte	Volume	Stock Concentration
As	400 µL	1000 ppm
Pb	200 µL	1000 ppm
Se	100 µL	1000 ppm
Tl	500 µL	1000 ppm
Cd	50 µL	1000 ppm

- Dilute to 100 mL with Type II water. This spike solution now contains:

4.0 ppm	Arsenic
2.0 ppm	Lead
1.0 ppm	Selenium
5.0 ppm	Thallium
0.5 ppm	Cadmium

Add 1000 µL of Spike Solution to water samples and 2000 µL to soil samples prior to digestion.

ICP
CLP-LOW SPIKES

- Add the analytes from the two tables below into separate 100 mL volumetric flasks which contain ~50 mL of Type II water and 5 mL of HNO_3.

ICP-1

Analyte	Volume	Stock Concentration	Spike Concentration
Sb	500 µL	1000 ppm	5 ppm
Al	5000 µL	1000 ppm	50 ppm
Ba	2000 µL	1000 ppm	20 ppm
Be	100 µL	1000 ppm	1 ppm
Cr	500 µL	1000 ppm	5 ppm
Co	1000 µL	1000 ppm	10 ppm
Cu	500 µL	1000 ppm	5 ppm
Fe	2500 µL	1000 ppm	25 ppm
Mn	500 µL	1000 ppm	5 ppm
Ni	1000 µL	1000 ppm	10 ppm
V	1000 µL	1000 ppm	10 ppm
Zn	1000 µL	1000 ppm	10 ppm

ICP-2

Analyte	Volume	Stock Concentration	Spike Concentration
Ag	500 µL	1000 ppm	5 ppm

- Add 1000 µL of ICP-1 and 1000 µL of ICP-2 spike solutions to water samples prior to digestion. For soil samples, add 2000 µL of each spike solution prior to digestion. The final digest will contain:

Analyte	Concentration in Digest	Analyte	Concentration in Digest
Sb	50 ppb	Al	500 ppb
Ba	200 ppb	Be	10 ppb
Cr	50 ppb	Co	100 ppb
Cu	50 ppb	Fe	250 ppb
Mn	50 ppb	Ni	100 ppb
V	100 ppb	Zn	100 ppb
Ag	50 ppb		

Table based on final volume of 100 mL for water digests, and 200 mL for soil digests.

GRAPHITE FURNACE
CLP-LOW SPIKES

- Add the analytes listed in the table below to a 100 mL volumetric flask which contains ~50 mL of Type II water.

Analyte	Volume	Stock Concentration	Spike Concentration
As	100 µL	1000 ppm	1000 ppb
Se	500 µL	1000 ppm	5000 ppb
Tl	500 µL	1000 ppm	5000 ppb
Pb	250 µL	1000 ppm	2500 ppb
Cd	100 µL	1000 ppm	1000 ppb

- Add 1000 µL of Spike Solution to water samples and 2000 µL to soil samples prior to digestion. The final digest will contain*:

10 ppb Arsenic
50 ppb Selenium
50 ppb Thallium
25 ppb Lead
10 ppb Cadmium

*Based on 100 mL final volume for water digests and 200 mL for soil digests.

ICP LCSW

The LCSW for the ICP digest was made by diluting 10 mLs of CLP-SPIKE and 50 mLs of LTL-6 solutions, and 2 mLs of conc. HNO_3 to 1000 mLs with type II water. A 100 mL aliquot of this solution was then digested.

ICP Analytes

Analyte	Concentration in Digest (ppb)	Analyte	Concentration in Digest (ppb)
Al	2000	Pb	500
Ba	2000	Mg	50000
Be	50	Mn	500
Cd	50	Ni	500
Ca	50000	K	50000
Cr	200	Ag	50
Co	500	Na	50000
Cu	250	V	500
Fe	1000	Zn	500
Sb	500		

Other Analytes

Analyte	Concentration in Digest (ppb)	Analyte	Concentration in Digest (ppb)
As	2000	Tl	2000
Se	2000		

Tables based on a final volume of 100 mL.

Graphite Furnace LCSW

- Add the analytes listed in the table below to a 100 mL volumetric flask which contains ~50 mL of Type II water and 2 mL of HNO_3. Dilute to volume with Type II water.

Analyte	Volume	Stock Concentration
As	500 µL	1000 ppm
Pb	500 µL	1000 ppm
Se	250 µL	1000 ppm
Tl	250 µL	1000 ppm
Cd	100 µL	1000 ppm

- Dilute to 100 mL with Type II water. This LCSW stock solution now contains:

5.0 ppm	Arsenic
5.0 ppm	Lead
2.5 ppm	Selenium
2.5 ppm	Thallium
1.0 ppm	Cadmium

The LCSW for the GFAA digest was made by diluting 10 mLs of LCSW stock solution and 20 mLs of conc. HNO_3 to 1000 mLs with type II water. A 100 mL aliquot of this solution was then digested.

Graphite Furnace Analytes

Analyte	Concentration in Digest (ppb)	Analyte	Concentration in Digest (ppb)
As	50	Tl	25
Pb	50	Cd	10
Se	25		

Tables based on a final volume of 100 mL.

PDCA
SPIKES and LCSW

The LCSW for the PDCA extraction was make by adding 10 mL SPEX ICV-1 solution to 100 mL of Type II water prior to extraction. The spike was made by adding 1000 µL of CLP SPK solution to a 100 mL aliquot of the sample prior to extraction.

ICP Analytes

Analyte	Concentration in Digest	Analyte	Concentration in Digest
Cd	50 ppb	Mn	500 ppb
Co	500 ppb	Ni	500 ppb
Cu	250 ppb	Ag	50 ppb
Fe	1000 ppb	V	500 ppb
Zn	500 ppb		

Graphite Furnace Analytes

Analyte	Concentration in Digest	Analyte	Concentration in Digest
Pb	500 ppb	Tl	2000 ppb
Se	2000 ppb		

Tables based on a final volume of 100 mL.

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

Method #: LTL-7010

Title: **Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FLAA or ICP Spectroscopy by SW846 Method 3010A**

Revision history:

Number	Date
1.	12/14/94
2.	06/03/96

UNCONTROLLED

Written by: Lucas Hawkins — Date: 6/4/96
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Reviewed by: Bill Lundberg — Date: 6/4/96
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Reviewed by: Harry Romberg — Date: 6-4-96
Harry Romberg, QC Officer

Approved by: Karen J. Kotz — Date: 6/4/96
Karen Kotz, Laboratory Director

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1. Introduction and Scope

1.1 This digestion procedure is used for the preparation of aqueous samples, TCLP and mobility-procedure extracts, and wastes that contain suspended solids for analysis by flame atomic absorption spectroscopy (FLAA) or inductively coupled argon plasma spectroscopy (ICP). The procedure is used to determine total metals.

1.2 Samples prepared by Method 3010A may be analyzed by FLAA or ICP for the following:

Aluminum	Calcium	Magnesium	*Silver
*Antimony (also GFAA)	Chromium	Manganese	Sodium
**Arsenic	Cobalt	Molybdenum	Thallium
Barium	Copper	Nickel	*Tin (FLAA only)
Beryllium	Iron	Potassium	Vanadium
Cadmium	Lead	**Selenium	Zinc

* Although Antimony, Silver and Tin are not listed in the original SW846 document as analytes that may be analyzed from samples prepared by Method 3010A, Laucks has demonstrated adequate recovery for these analytes from this digestate. In addition, Laucks has demonstrated adequate Antimony recovery in samples prepared by LTL-7010 and analyzed by GFAA.

** Analysis by ICP

1.3 With the exception of Antimony, this digestion procedure is not suitable for samples which will be analyzed by graphite furnace atomic absorption spectroscopy because hydrochloric acid can cause interference during furnace atomization. Consult Method LTL-7008 for samples requiring graphite furnace analysis.

2. Summary of Method

2.1 A mixture of nitric acid and the material to be analyzed is refluxed in a covered beaker. This step is repeated with additional portions of nitric acid until the digestate is light in color or until its color has stabilized. After the digestate has been brought to a low volume, it is refluxed with hydrochloric acid and brought up to volume. If a sample goes to dryness, it must be discarded and prepared again.

3. Safety Procedures

3.1 These procedures involve the use of strong and/or hot acid solutions. The analyst must wear eye protection, a lab coat, and gloves to protect against burns.

3.2 These procedures involve hot plates which may present the danger of burns from heated surfaces and electrical hazards. The analyst must take appropriate caution to avoid injury from these sources.

3.3 Samples and spiking solutions may contain high levels of toxic metals and other unknown constituents. The analyst must take every precaution to avoid contact with these potentially hazardous materials and should wash hands thoroughly before eating or going home for the day.

4. Equipment

- 150-mL beakers or equivalent, acid-washed
- 100-mL graduated cylinder, acid-washed
- Ribbed watch glasses, acid-washed
- Eppendorf or other micropipets
- Hot plate or equivalent heating source, one that is adjustable and capable of maintaining a temperature of 90-95° C.
- Glass funnels, acid-washed
- Filter paper, Whatman No. 41 or equivalent
- Sample digestate bottles, acid-washed
- Thermometer

Note: All glassware used for this digestion must be prepared by the method stated in Inorganics Glass Cleaning Procedures, LTL-7003.

5. Reagents

5.1 Reagent Water. Reagent water will be interference-free. All references to water in the method refer to reagent water unless otherwise specified.

5.2 Nitric acid (concentrated), HNO_3, ACS Reagent grade or better.

5.3 Hydrochloric acid (concentrated), HCl, ACS Reagent grade or better.

The method blank, prepared along with the samples, is used as a contamination check. Since the holding time for all analytes associated with this method have a holding time of 6 months, repreparation would not be an issue if contamination was traced to a specific reagent.

6. Acid Digestion Procedure

See Appendix A for digestion log.
See Appendix B for flowchart.
See Appendix C for Quality Control solutions.

6.1 Transfer a 100-mL representative aliquot of the well-mixed sample to a 150-mL beaker and add 3 mL of concentrated HNO_3. If only one graduated cylinder is used for all samples, the preparation blank is to be the last sample dispensed to assure the absence of sample carry-over. Cover the beaker with a ribbed watch glass. Place the beaker on a hot plate and carefully evaporate to 5 mL, making certain that the sample does not boil and that no portion of the bottom of the beaker is allowed to go dry. Place a thermometer into a beaker of water on the hotplate. Record the temperature achieved during the digest on the digest log. Cool the beaker and add another 3-mL aliquot of HNO_3. Return the covered beaker to the hot plate and adjust the temperature, if necessary, so that a gentle reflux action occurs.

6.2 Continue heating, adding additional HNO_3 as necessary until the digestion is complete, indicated when the digestate is light in color or does not change in appearance upon additional heating. Evaporate the samples to 3 mL, not allowing any portion of the bottom of the beaker to go dry. After allowing the beakers to cool, add 5 mL of concentrated HCl and 5 mL reagent water. Reflux the samples for an additional 15 minutes to dissolve any precipitate or residue resulting from evaporation.

Note: If a sample is allowed to go to dryness at any stage of the digestion procedure, low recoveries will result. The sample must be discarded and prepared again.

6.3 After cooling the beakers, add 5mL concentrated HCl to obtain a final acid concentration of approximately 10%. Pour each sample into a 100-mL graduated cylinder, rinsing the beaker walls with water and adding the rinsate to the graduated cylinder, and bring to a final volume of 100 mL. If one graduated cylinder is to be used to adjust volume of each sample, the blank is to be the last sample dispensed. If silicates or other insoluble material that could clog the nebulizer are present in the samples, the samples should be allowed to settle overnight prior to analysis. Filtration should be performed only if there is concern that the insoluble material will not settle out of solution. Filter and dilute the samples to a final volume of 100 mL, using acid-washed filter apparatus to avoid sample contamination. Samples should be stored in acid-washed digestate bottles.

6.4 The prepared samples are then transferred to the metals analysis department. The digestion log is to reflect the time at the start and finish of the digest. In order to maintain strict chain of custody, the time and date when the digestates are relinquished to the analysis department, as well as the initials of the analyst accepting the digest, are recorded on the digestion log.

7. Quality Control

7.1 In each batch or SDG consisting of no more than 20 samples, a preparation blank is created. This consists of 100 mL of reagent water and the appropriate amounts of nitric and hydrochloric acid, digested in exactly the same way as the samples. If only one graduated cylinder is used for all samples, the preparation blank is to be the last sample

dispensed to assure the absence of sample carry-over. The blank should be labeled on the digestate bottle in the following way: B, date, instrument, S or W (for soil or water) and the sequence number of the digestion. Example: B111794ICPW01.

7.2 Spiked samples should be employed to determine accuracy. In each batch or SDG consisting of no more than 20 samples, either matrix spike / sample duplicate (MS/Dup) or matrix spike / matrix spike duplicate (MS/MSD) samples will be prepared. For SW846-specified quality control measures, MS/Dup samples will be prepared in the following way. Dispense one sample in triplicate. Two will be digested exactly as any other sample. The third must be spiked with the appropriate spike solutions determined by the analyst, and then treated as any other sample. All glassware and digestate bottles must be marked appropriately. If MS/MSD samples are required instead of the SW846-specified MS/Dup, both the second and third aliquots must be spiked and all glassware and digestate bottles marked appropriately.

7.3 Determine the analytes required for analysis by consulting sample work order information. Each spiking solution is given its own unique number according to the page and line of the standards logbook which it is entered into. This number and the volume dispensed must be clearly recorded on the digestion log. A sample page from the standards logbook is included in Appendix C.

7.4 In each batch or SDG consisting of no more than 20 samples, either a Blank Spike (BLK SPK) or a Laboratory Control Sample (ICP LCSW) must be digested in exactly the same manner as a sample. A Blank Spike consists of 100 mL of reagent water spiked at the levels determined by the analyst as outlined in Section 7.3.

8. References

8.1 USEPA, Test Methods for Evaluating Solid Waste Physical/Chemical Methods, USEPA SW846, most recent version, Method 3010A.

Appendix A, Sample LTL-7010 Digestion Logs

LAUCKS TESTING LABORATORIES, INC.					ICP DIGESTION LOG				Place a √ in the box next to the method performed.			
SDG #:	Digest start : date/time				Digest finish : date/time				SW-846 3010A		CLP-water	
Analyst:	/				/				SW-846 3050A		CLP-soil	

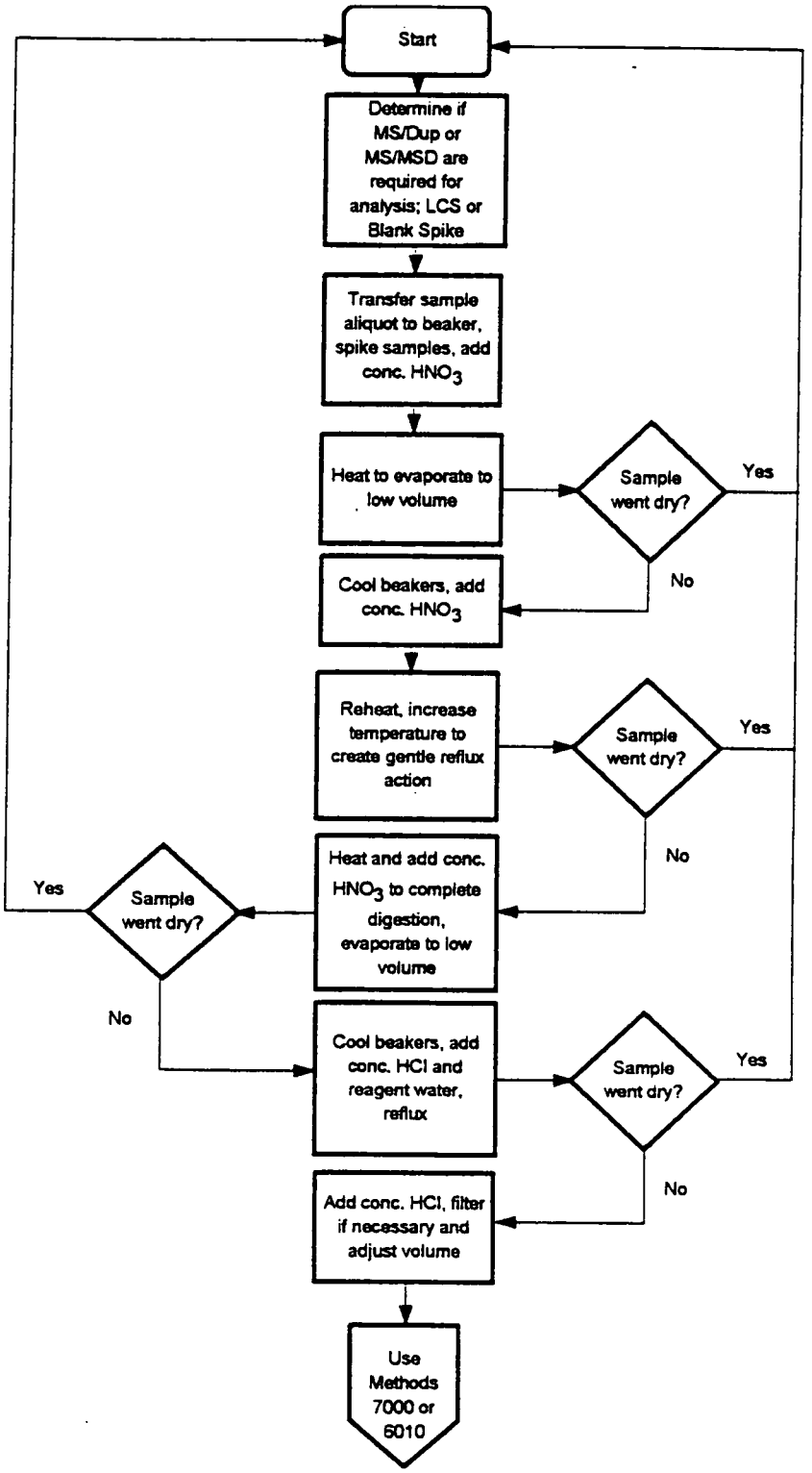
Sample ID	Sample Size (g:mL)	Final Volume (mL)	Color		Clarity		Text	Art	Comments
			Bef	Aft	Bef	Aft			

Chain of Custody	Digest Relinquished Date / Time : Analyst	Digest Accepted Analyst	Digest Disposed Date	Hot Plate Temperature, °C
	/			

Color 1=Red, 2=Blue, 3=Yellow, 4=Green, 5=Orange, 6=Violet, 7=White, 8=Brown, 9=Grey, 10=Black, 11=Colorless
Clarity 1=Clear, 2=Cloudy, 3=Opaque
Texture F=Fine (powdery), M=Medium (sandy), C=Coarse (rocky) Artifacts: If "Yes", give description in the comments field.

P:\METALS\BNCHSHEET\DIG.D

Appendix B, Flowchart for LTL-7010


Start
Determine if MS/Dup or MS/MSD are required for analysis; LCS or Blank Spike
Transfer sample aliquot to beaker, spike samples, add conc. HNO_3
Heat to evaporate to low volume
Sample went dry?
Yes
No
Cool beakers, add conc. HNO_3
Reheat, increase temperature to create gentle reflux action
Sample went dry?
Yes
No
Heat and add conc. HNO_3 to complete digestion, evaporate to low volume
Sample went dry?
Yes
No
Cool beakers, add conc. HCl and reagent water, reflux
Sample went dry?
Yes
No
Add conc. HCl, filter if necessary and adjust volume
Use Methods 7000 or 6010

Appendix C, Quality Control Solutions

ICP LCSW

The LCSW for the ICP digest was made by diluting 100 mLs of SPEX ICV-1™ solution to 1000 mLs with type II water. A 100 mL aliquot of this solution was then digested.

ICP Analytes

Analyte	Concentration in Digest (ppb)	Analyte	Concentration in Digest (ppb)
Al	20000	Pb	500
Ba	20000	Mg	500000
Be	500	Mn	1500
Cd	500	Ni	4000
Ca	500000	K	500000
Cr	1000	Ag	1000
Co	5000	Na	500000
Cu	2500	V	5000
Fe	10000	Zn	2000
Sb	6000		

Other Analytes

Analyte	Concentration in Digest (ppb)	Analyte	Concentration in Digest (ppb)
As	1000	Tl	1000
Se	500		

Tables based on a final volume of 100 mL.

ICP Spikes

- The CLP spike solution for ICP digest was made by diluting 10 mLs of CLPP-SPK-1™, 10 mLs of CLPP-SPK-2™, and 10 mLs of CLPP-SPK-3™ to 100 mLs with Type II water.

- Add 1000 µLs of CLP spike solution to water samples and 2000 µLs of CLP spike solution to soil samples prior to digestion.

ICP Analytes

Analyte	Concentration in Digest (ppb)	Analyte	Concentration in Digest (ppb)
Ag	50	Fe	1000
Al	2000	Mn	500
As	2000	Ni	500
Ba	2000	Pb	500
Be	50	Se	2000
Cd	50	Sb	500
Co	500	Tl	2000
Cr	200	V	500
Cu	250	Zn	500

Table based on a final volume of 100 mL for water digests, and 200 mL for soil digests.

ICP
CLP-LOW SPIKES

- Add the analytes from the two tables below into separate 100 mL volumetric flasks which contain ~50 mL of Type II water and 5 mL of HNO_3.

ICP-1

Analyte	Volume	Stock Concentration	Spike Concentration
Sb	500 µL	1000 ppm	5 ppm
Al	5000 µL	1000 ppm	50 ppm
Ba	2000 µL	1000 ppm	20 ppm
Be	100 µL	1000 ppm	1 ppm
Cr	500 µL	1000 ppm	5 ppm
Co	1000 µL	1000 ppm	10 ppm
Cu	500 µL	1000 ppm	5 ppm
Fe	2500 µL	1000 ppm	25 ppm
Mn	500 µL	1000 ppm	5 ppm
Ni	1000 µL	1000 ppm	10 ppm
V	1000 µL	1000 ppm	10 ppm
Zn	1000 µL	1000 ppm	10 ppm

ICP-2

Analyte	Volume	Stock Concentration	Spike Concentration
Ag	500 µL	1000 ppm	5 ppm

- Add 1000 µL of ICP-1 and 1000 µL of ICP-2 spike solutions to water samples prior to digestion. For soil samples, add 2000 µL of each spike solution prior to digestion. The final digest will contain:

Analyte	Concentration in Digest	Analyte	Concentration in Digest
Sb	50 ppb	Al	500 ppb
Ba	200 ppb	Be	10 ppb
Cr	50 ppb	Co	100 ppb
Cu	50 ppb	Fe	250 ppb
Mn	50 ppb	Ni	100 ppb
V	100 ppb	Zn	100 ppb
Ag	50 ppb		

Table based on final volume of 100 mL for water digests, and 200 mL for soil digests.

PDCA
SPIKES and LCSW

The LCSW for the PDCA extraction was make by adding 10 mL SPEX ICV-1™ solution to 100 mL of Type II water prior to extraction. The spike was made by adding 1000 µL of CLP SPK solution to a 100 mL aliquot of the sample prior to extraction.

ICP Analytes

Analyte	Concentration in Digest	Analyte	Concentration in Digest
Cd	50 ppb	Mn	500 ppb
Co	500 ppb	Ni	500 ppb
Cu	250 ppb	Ag	50 ppb
Fe	1000 ppb	V	500 ppb
Zn	500 ppb		

Graphite Furnace Analytes

Analyte	Concentration in Digest	Analyte	Concentration in Digest
Pb	500 ppb	Tl	2000 ppb
Se	2000 ppb		

Tables based on a final volume of 100 mL.

LAUCKS TESTING LABORATORIES, INC.
STANDARDS LOGBOOK

#	ANALYTE	ID or STOCK No.	STOCK CONC.	VOL/WT TAKEN	FINAL VOL.	CONC.	SOLVENT	PREP. DATE	INIT.	EXP. DATE	QC'D (Initials)
1	CLP.SPK	ME1.03.06 EXP 12/12/96	(CLPP.SPK 1)	10mL	100mL		5% HNO_3 H_2O	03/28/96	RJH	12/12/96	
		ME1.03.07 EXP 12/12/96	(CLPP.SPK 2)								
		ME1.03.01 EXP 12/12/96	(CLPP.SPK 3)								
2	GF-CAL1 (Pb)	ME3.86.09 EXP. 7/10/96	1000 S	30µL	100mL	0.3 ppm	0.5% HNO_3 H_2O	03/28/96	RJH	05/30/96	
	(Se)	ME3.86.10 EXP 7/10/96		50µL		0.5 ppm					
	(As)	ME3.81.9 EXP 5/30/96		100µL		1.0 ppm					
	(Tl)	ME3.86.11 EXP 7/10/96		100µL		1.0 ppm					
3	GF-CAL2 (Pb)	ME3.86.09 EXP 7/10/96	1000 S	100µL	100mL	1.0 ppm	0.5% HNO_3 H_2O	03/28/96	RJH	05/30/96	
	(Se)	ME3.86.10 EXP 7/10/96		100µL		1.0 ppm					
	(As)	ME3.81.9 EXP 5/30/96		250µL		2.5 ppm					
	3/28/96 RJH										

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP: LTL-7012

Title: **Microwave Assisted Acid Digestion of Sediments, Sludge, Soils, and Oils Using SW846 Method 3051**

Revision history:

Number	Date
0.	6/04/96
1.	8/21/98

UNCONTROLLED

Revised by: _____ Date: 08-21-98
Ming-Hwa Hwang, Metals Supervisor

Reviewed by: Harry Romberg Date: 8-21-98
Harry Romberg, QA Officer

Approved by: Kathy Kreps Date: 8-24-98
Kathy Kreps, Laboratory Director

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1. Introduction and Scope

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

1.2 This digestion procedure is used for the preparation of sediments, sludge, soils, and oils for analysis by flame atomic absorption spectroscopy (FLAA), graphite furnace absorption spectroscopy (GFAA), inductively coupled argon plasma spectroscopy (ICP), or inductively coupled argon plasma mass spectrometry (ICP-MS). The procedure is a hot acid leach for determination of available metals.

1.3 Samples prepared by Method 3051 using nitric acid digestion may be analyzed by FLAA, GFAA, ICP or ICP-MS for the following:

Aluminum	Cadmium	Iron	Molybdenum	Sodium
Antimony	Calcium	Lead	Nickel	Strontium
Arsenic	Chromium	Magnesium	Potassium	Thallium
Boron	Cobalt	Manganese	Selenium	Vanadium
Barium	Copper	Mercury	Silver	Zinc
Beryllium				

1.4 This method is provided as an alternate to LTL-7011. It is intended to provide a rapid multielement acid leach digestion prior to analysis by Flame Atomic Absorption(FLAA), Graphite Furnace Atomic Absorption(GFAA), Inductively Coupled Plasma Emission Spectroscopy, or Inductively Coupled Plasma Mass Spectroscopy(ICP-MS). If a decomposition including Hydrochloric Acid is required for certain elements, it is recommended that LTL-7011 be used. The parameters which are quoted in this procedure are those recommended by the manufacturer of the microwave digestion system used, in order to be in compliance with SW-846 protocols.

2. Summary of Method

2.1 A representative sample of up to 0.5g is digested in a Teflon vessel with 10 mL of HNO_3 in a microwave unit. After cooling, the vessel contents are either filtered or allowed to settle and diluted to volume and analyzed by an appropriate method.

3. Safety Procedures

3.1 These procedures involve the use of strong and/or hot acid solutions. The analyst must wear eye protection, a lab coat, and gloves to protect against burns.

3.2 Samples and spiking solutions may contain high levels of toxic metals and other unknown constituents. The analyst must take every precaution to avoid contact with these potentially hazardous materials and should wash hands thoroughly before eating or leaving the laboratory.

3.3 **Very reactive or volatile materials may create high pressures when heated, causing the vessels to vent with potential loss of sample and analytes. Samples that contain carbonates or other carbon dioxide generating compounds may generate enough pressure to vent the vessel. If this situation is anticipated the analyst may wish to use a smaller sample or an alternative digestion method.**

4. Equipment

- MDS-2100 Microwave Digestion system, CEM
- 120 mL Teflon® vessels and caps, carousel
- 50 or 100 mL graduated cylinder, acid washed
- Glass funnels, acid-washed
- Filter paper, Whatman No. 41 or equivalent
- Sample digestate bottles, acid washed
- Analytical balance, capable of weighing ± 0.01 g

Note: All glassware used for this digestion must be prepared by the method stated in Inorganics Glassware Cleaning Procedures, LTL-7003.

All Teflon® vessels and caps are to be thoroughly rinsed with deionized water prior to digestions and shaken to remove any significant droplets in the vessels.

The microwave is to be calibrated according to the procedures stated in SW846 method 3051. The MDS-2100 microwave, being designed with temperature control, does not require the calibration outlined in method 3051.

5. Reagents

5.1 Reagent Water - Reagent water will be interference free. All references to water in the method refer to reagent water unless otherwise specified.

5.2 Nitric acid (concentrated), HNO_3, ACS Reagent grade or better. Acid should be analyzed to determine levels of impurities. If the method blank is less than reporting limit then the acid can be used.

6. Microwave Sample Digestion Procedure

See Appendix A for digestion log
See Appendix B for flowchart
See Appendix C for quality control solutions

6.1 Weigh a well mixed sample directly into the Teflon digestion vessel to the nearest 0.01g. For soils, sludge and sediments, use no more than 0.50g. For oils, use no more than 0.25g.

6.2 Add 10 mL of concentrated nitric acid to each vessel and cap hand tight. If a vigorous reaction occurs with the addition of the acid, wait for the reaction to subside before capping. For each microwave digestion run, the pressure control lid is to accompany the sample that seems to have the most carbon. Weigh each capped vessel to the nearest 0.01 g.

6.3 Microwave

6.3.1 MDS-2100 Microwave Digestion system, CEM

6.3.2 Place the carousel in the unit and attach the pressure control line to the digestion vessel with the double ported lid. Run the heating program SW-3051 (temperature).

Program SW-3051 (temperature) as follows:

Stage	(1)	(2)	(3)	(4)	(5)
%POWER	100	0	0	0	0
PSI	70	0	0	0	0
RAMP	05:00	0000	0000	0000	0000
HOLD	04:30	0000	0000	0000	0000
TEMP	175	000	000	000	000

6.4 At the end of the microwave program, allow the vessels to cool in the unit for a minimum of 5 minutes before removal to avoid possible injury if a vessel vents immediately after microwave heating. Once they are at room temperature, weigh and record the weight of each vessel assembly. If the weight of the sample plus acid has decreased by more than 10%, discard and prepare the sample again.

6.5 Uncap the vessels in a fume hood due to the likely presence of nitrogen oxide. Dilute sample to 50 mL in a graduated cylinder and transfer the samples to an acid-cleaned polyethylene bottle. If only one graduated cylinder is used to volume all samples, the blank is to be the final sample diluted to assure the absence of sample carry-over. If the digested sample contains particulate which may clog nebulizers or interfere with injection of the sample into an instrument, the sample may be allowed to settle or be filtered.

6.6 The prepared samples are then transferred to the metals analysis department. The digestion log is to reflect the time at the start and finish of the digest. In order to maintain a strict chain of custody, the time and date when the digestates are relinquished to the analysis department, as well as the initials of the analyst accepting the digest, are recorded on the digestion log

7. Quality Control

7.1 In each batch or SDG consisting of no more than 20 samples, a preparation blank is created. The blank should be labeled on the digestate bottle in the following way: B, date, M (microwave), S (soil) and the sequence number of the digestion. Example B061598MS01.

7.2 Spiked samples should be employed to determine accuracy. In each batch or SDG consisting of no more than 20 samples, either matrix spike / sample duplicate (MS/Dup) or matrix spike /matrix spike duplicate (MS/MSD) samples will be prepared. For SW3051-specified quality control measures, MS/Dup samples will be prepared in the following way. Dispense a sample in triplicate. Two will be analyzed exactly as any other sample. The third must be spiked with the appropriate spike solutions, determined by the analyst. All glassware and digestate bottles must be marked appropriately. If MS/MSD samples are required instead of the SW846-specified MS/Dup, both the second and third aliquot must be spiked.

7.3 Determine the analytes required for analysis by consulting sample work order information. The analyst must record the specific spiking solution(s) used, the volume(s) of the solution(s), and the corresponding Metals Standard Logbook number(s) on the digestion log as illustrated in Appendix C

7.4 In each batch or SDG consisting of no more than 20 samples, prepare a Lab Control Sample (LCSS) in exactly the same manner as a sample. All spikes and standard reference materials must be recorded by the analyst on the digestion log. The record must state the specific spiking solution used, the volume of the solution, and it's Metals Standard Logbook number, and the lot number of any reference materials.

8. References

8.1 USEPA, Test Methods for Evaluating Solid Waste Physical/Chemical Methods, USEPA SW846, most recent version, Method 3051.

Appendix A, Sample LTL-7012 Digestion Logs

Laucks Testing Laboratories, INC.	START DATE TIME	FINISH DATE TIME	METHOD	ANALYST	ANALYSIS
SDG #:			SW846 3051		Microwave Digestion
MATRIX: SOIL					

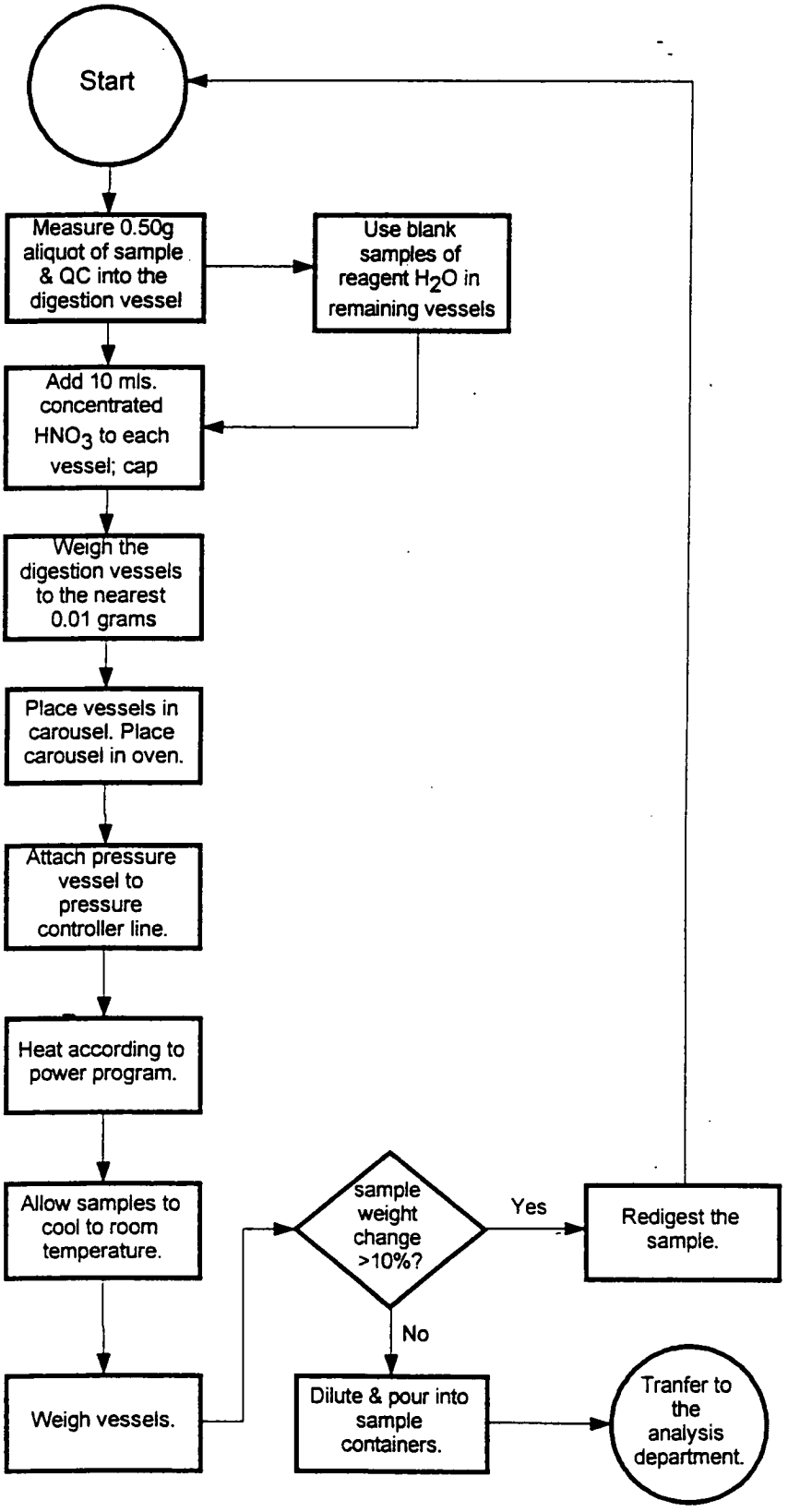
Bomb #	Sample ID	Samp (g)	Final Vol (mL)	Color		Text	Artifacts	Bomb & Sample (g)	Final Bomb (g)	Δ (g)	Δ >0.5 g?
				Int	Fin						

Chain of Custody	Digest Relinquished Date / Time : Analyst	Digest Accepted :Analyst	Digest Disposed :Date
	/ :		

COLOR: 1 = Red, 2 = Blue, 3 = Yellow, 4 = Green, 5 = Orange, 6 = Violet, 7 = White, 8 = Brown, 9 = Gray, 10 = Black, 11 = Colorless.

TEXTURE: F = Fine (Powdery), M = Medium (Sandy), C = Coarse (Rocky).

Appendix B, Flowchart for LTL-7012


Start
Measure 0.50g aliquot of sample & QC into the digestion vessel
Use blank samples of reagent H_2O in remaining vessels
Add 10 mls. concentrated HNO_3 to each vessel; cap
Weigh the digestion vessels to the nearest 0.01 grams
Place vessels in carousel. Place carousel in oven.
Attach pressure vessel to pressure controller line.
Heat according to power program.
Allow samples to cool to room temperature.
Weigh vessels.
sample weight change >10%?
Yes
Redigest the sample.
No
Dilute & pour into sample containers.
Tranfer to the analysis department.

Appendix C, Quality Control Solutions

ICP Spikes

- The CLP spike solution for ICP digest was made by diluting 10 mLs of CLPP-SPK-1™, 10 mLs of CLPP-SPK-2™, and 10 mLs of CLPP-SPK-3™ to 100 mLs with Type II water.

- Add 500 µLs of CLP spike solution to soil samples prior to digestion.

ICP Analytes

Analyte	Concentration in Digest (µg/L)	Analyte	Concentration in Digest (µg/L)
Ag	50	Fe	1000
Al	2000	Mn	500
As	2000	Ni	500
Ba	2000	Pb	500
Be	50	Se	2000
Cd	50	Sb	500
Co	500	Tl	2000
Cr	200	V	500
Cu	250	Zn	500

Table based on a final volume of 50 mL for soil digests.

GRAPHITE FURNACE SPIKES

- Add the analytes listed in the table below to a 100 mL volumetric flask which contains ~50 mL of Type II water and 5 mL of HNO_3.

Analyte	Volume	Stock Concentration
As	400 µL	1000 mg/L
Pb	200 µL	1000 mg/L
Se	100 µL	1000 mg/L
Tl	500 µL	1000 mg/L

- Dilute to 100 mL with Type II water. This spike solution now contains:

4.0 mg/L	Arsenic
2.0 mg/L	Lead
1.0 mg/L	Selenium
5.0 mg/L	Thallium

- Add 500 µL of Spike Solution to soil samples prior to digestion.

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

Method #: LTL-7015

Title: **Acid Digestion of Sediments, Sludge, and Soils by SW846 Method 3050B** .

Revision history:

Number	Date
0.0	1/18/99
1.0	3/20/00

Controlled Document
No. 20 Assigned to: Tetra

Revised by: Stan Dittrick — Date: 3/20/00
Stan Dittrick, Chemist

Reviewed by: Paul Swift — Date: 03/21/2000
Paul Swift, Inorganic Division Manager

Reviewed by: Harry Romberg — Date: 3-20-2000
Harry Romberg, QA Officer

Written by: Kathy Kreps — Date: 03-22-2000
Kathy Kreps, Laboratory Director

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1. Introduction and Scope

1.1. This method is an acid digestion procedure used to prepare sediments, sludges, and soil samples for analysis by flame or furnace atomic absorption spectroscopy (FLAA and GFAA, respectively) or by inductively coupled argon plasma spectroscopy (ICP) or inductively coupled argon plasma spectroscopy - mass spectrometry (ICP-MS). Samples prepared by this method may be analyzed by ICP for all the listed metals, or by FLAA or GFAA as indicated below (see also step 2.1):

FLAA/ICP-AES		GFAA/ICP-MS
Aluminum	Magnesium	Arsenic
Antimony	Manganese	Beryllium
*Arsenic	Molybdenum	Cadmium
Barium	Nickel	Chromium
Beryllium	Potassium	Cobalt
Cadmium	*Selenium	Iron
Calcium	Silver	Lead
Chromium	Sodium	Molybdenum
Cobalt	Thallium	Selenium
Copper	*Tin (FLAA only)	*Tin (ICP-MS only)
Iron	Vanadium	Thallium
Lead	Zinc	

* Although Arsenic and Selenium (analyzed by FLAA/ICP) and Tin (analyzed by FLAA or ICP-MS) are not listed in the original SW846 document as analytes that may be analyzed from samples prepared by Method 3050B, Laucks has demonstrated adequate recovery for these analytes from this digestate.

2. Summary of Method

2.1 A representative 1. to 1.5g sample is digested in nitric acid and hydrogen peroxide. The digestate is then either reduced to a low volume or refluxed with hydrochloric acid. Hydrochloric acid is used for flame AA and ICP analyses only. Dilute hydrochloric acid is used as the final reflux acid for (1) the ICP analysis of As and Se, (2) the flame AA or ICP analysis of Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, Pb, Sb, Sn, Tl, V, and Zn. The hydrochloric acid step is omitted for the furnace AA analysis of As, Be, Cd, Cr, Co, Fe, Pb, Mo, Se, and Tl. The diluted samples have an approximate acid concentration of 5% (v/v).

3. Safety Procedures

3.1 These procedures involve the use of strong and/or hot acid solutions. The analyst must wear eye protection, a lab coat, and gloves to protect against burns.

3.2. These procedures involve hot plates which may present the danger of burns from heated surfaces and electrical hazards. The analyst must take appropriate caution to avoid injury from these sources.

3.3. Samples and spiking solutions may contain high levels of toxic metals and other unknown constituents. The analyst must take every precaution to avoid contact with these potentially hazardous materials and should wash hands thoroughly before taking any breaks, eating, or going home for the day.

3.4. Fertilizer samples are chemically complex matrices and should be handled with extreme caution. Many components found in fertilizer are highly reactive to acid and all acid additions should be made with special care, in a hood, standing back from the sample. Also, these samples have been known to "pop" or explode during heating, so care should be taken not to stand close to or have any body part over the samples while they are being heated.

4. Equipment

- 250-mL Erlenmeyer flasks or equivalent, acid-washed
- Analytical balance capable of accurately weighing to the nearest 0.01 g
- Reflux caps fitted for Erlenmeyer flasks, acid-washed
- Eppendorf or other micropipets
- Hot plate or equivalent heating source, adjustable and capable of maintaining a temperature of 90-95° C
- 100-mL graduated cylinder, acid-washed

- Filter paper, Whatman No. 41 or equivalent
- Glass funnels, acid-washed
- 100-mL volumetric flasks, acid-washed
- Sample digestate bottles, acid-washed
- Thermometer

Note: All glassware used for this digestion must be prepared by the method stated in Inorganics Glass Cleaning Procedures, SOP #: LTL-7003.

5. Reagents

5.1 Reagent Water. Reagent water will be interference-free deionized water. All references to water in the method refers to reagent water unless otherwise specified.

5.2 Nitric acid (concentrated), HNO_3, ACS Reagent grade or better

5.3 Hydrochloric acid (concentrated), HCl, ACS Reagent grade or better

5.4 Hydrogen peroxide (30%), H_2O_2, ACS Reagent grade or better

The method blank prepared along with the samples is used as a contamination check. Since the holding time for all analytes associated with this method have a holding time of 6 months, repreparation would not be an issue if contamination was traced to a specific reagent.

6. Acid Digestion Procedure

See Appendix A for digestion logs.
See Appendix B for flowchart.
See Appendix C for Quality Control solutions
See Appendix D for Procedure Checklist

6.1 Mix the sample thoroughly to achieve homogeneity. For each digestion procedure, weigh 1.00-1.50 g of sample to the nearest 0.01 g directly into an Erlenmeyer flask. For samples with low percent solids, a larger sample size may be used to attain the equivalent of 1.00-1.50 g dry basis as long as digestion is completed. Add appropriate spiking mix aliquots to QC samples at this point.

6.2 Place the reflux caps on the flasks, add 5 mL of concentrated HNO_3, 5 mL H_2O and mix the slurry. Heat the samples to 95° C and reflux without boiling for 15 minutes. Allow the samples to cool. Record the temperature in the digest log. Add 5 mL of concentrated HNO_3, heat 30 min and allow the samples to cool. If brown fumes are generated continue to add

acid, heat and cool until fumes are no longer emitted. Reflux the samples for two hours or allow them to evaporate to 5 mL with reflux caps. Allow the samples to cool.

6.3 Add 2 mL water, 3 mL 30% H_2O_2, heat for 10 minutes and cool the samples. Add 1 mL 30% H_2O_2, reflux for 10 minutes and allow the samples to cool. Repeat the addition of 1 mL H_2O_2 and heat until effervescence is minimal or a maximum of 10 mL H_2O_2 is added. Make sure that sample losses do not occur due to excessive effervescence. Reduce the sample to 5 mL.

6.4 If the sample is being prepared for GFAA or ICP-MS analysis then skip this step otherwise add 10 mL of concentrated HCl. Heat the sample for 15 minutes and allow it to cool.

6.5 Dilute the sample to 100 mL with water in a graduated cylinder and place in an acid-washed digestate bottle. If only one graduated cylinder is to be used to measure all samples, then bring the blank to volume after the rest of the samples. This will help to determine if their is any sample carry over contamination.

Note: If a sample is allowed to go dry at any stage of the digestion procedure, the sample must be discarded and redigested.

6.6 If silicates or other insoluble material that could clog the nebulizer or the graphite furnace autosampler are present in the samples, allow the samples to settle overnight prior to analysis. If there is concern that the insoluble material will not settle out of solution filter the sample with Whatman No. 41 or equivalent filter paper.

6.7 In the digestion log record the start date/time, finish date/time, and the date/time digestates are relinquished. Transfer the samples to the metals analysis department. Have the analyst accepting the digestates initial and date the digestion log

7. Quality Control

7.1 In each batch or SDG consisting of no more than 20 samples, a preparation blank is created. This consists of an empty, acid-washed Erlenmeyer flask to which the appropriate reagents are added and digested in exactly the same manner as a sample The blank should be labeled on the digestate bottle in the following way: B, date, instrument, S or W (for soil or water) and the sequence number of the digestion. Example: B111794ICPS01.

7.2 Spiked samples should be employed to determine accuracy. In each batch or SDG consisting of no more than 20 samples, either matrix spike / sample duplicate (MS/Dup) or matrix spike / matrix spike duplicate (MS/MSD) samples will be prepared. For SW846-

specified quality control measures, MS/Dup samples will be prepared in the following way. Dispense one sample in triplicate. Two will be digested exactly as any other sample. The third must be spiked with the appropriate spike solutions determined by the analyst, and then treated as any other sample. All glassware and digestate bottles must be marked appropriately. If MS/MSD samples are required instead of the SW846-specified MS/Dup, both the second and third aliquots must be spiked and all glassware and digestate bottles marked appropriately. See Appendix C for instructions on the preparation and use of QC solutions. Spiking levels presented are to be used only as guidance. The actual analytes and concentrations may vary.

7.3 Determine the analytes required for analysis by consulting sample work order information. Each spiking solution is given its own unique number according to the page and line of the standards logbook which it is entered into. This number and the volume dispensed must be clearly recorded on the digestion log. A sample page from the standards logbook is included in the Appendix C.

7.4 In each batch or SDG consisting of no more than 20 samples, either a Blank Spike (BLK SPK) or a Laboratory Control Sample (ICP or GF LCSS) must be digested in exactly the same manner as a sample. A Blank Spike consists of an empty, acid-washed Erlenmeyer flask to which the appropriate spikes (determined as outlined in Section 7.3) and reagents are added. The Certified Value and Advisory range for each analyte in a particular LCSS can be accessed from the Quality Control Database.

8. References

8.1 USEPA, Test Methods for Evaluating Solid Waste Physical/Chemical Methods, USEPA SW846, most recent version, Method 3050B.

Appendix A, LTL-7015 Digestion Logs

LAUCKS TESTING LABORATORIES, INC.		ICP DIGESTION LOG	Place a √ in the box next to the method performed.			
	Digest start : date/time	Digest finish : date/time	SW-846 3005A		ILM04.0-water	
SDG #:			SW-846 3010A		ILM04.0-soil	
Analyst:			SW-846 3050A		SW-846 3050B	

	Sample ID	Sample Size (g:mL)	Final Volume (mL)	Color		Clarity		Text	Art	Comments	Bottle #
				Bef	Aft	Bef	Aft				
01											
02											
03											
04											
05											
06											
07											
08											
09											
10											
11											
12											
13											
14											
15											
16											
17											
18											
19											
20											
21											
22											
23											
24											

Chain of Custody	Digest Relinquished Date / Time : Analyst	Digest Accepted Analyst	Digest Disposed Date	Hot Plate Temperature, °C
Reagents Lot Number	HNO_3	HCl	H_2O_2	

Color 1=Red, 2=Blue, 3=Yellow, 4=Green, 5=Orange, 6=Violet, 7=White, 8=Brown, 9=Grey, 10=Black, 11=Colorless
Clarity 1=Clear, 2=Cloudy, 3=Opaque
P:\metals\bnchshee\icpdig.doc
Texture F=Fine (powdery), M=Medium (sandy), C=Coarse (rocky) Artifacts: If "Yes", give description in the comments field.

P:\metals\bnchshee\icpdig.doc

LAUCKS TESTING LABORATORIES, INC.		GRAPHITE FURNACE DIGESTION LOG	Place a √ in the box next to the method performed.		
	Digest start : date/time	Digest finish : date/time	SW-846 3020A		ILM04.0-water
SDG #:			SW-846-7060A		ILM04.0-soil
Analyst:			SW-846 3050A		SW-846 3050B

	Sample ID	Sample Size (g:mL)	Final Volume (mL)	Color		Clarity		Text	Art	Comments	Bottle #
				Bef	Aft	Bef	Aft				
01											
02											
03											
04											
05											
06											
07											
08											
09											
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21											
22											
23											
24											

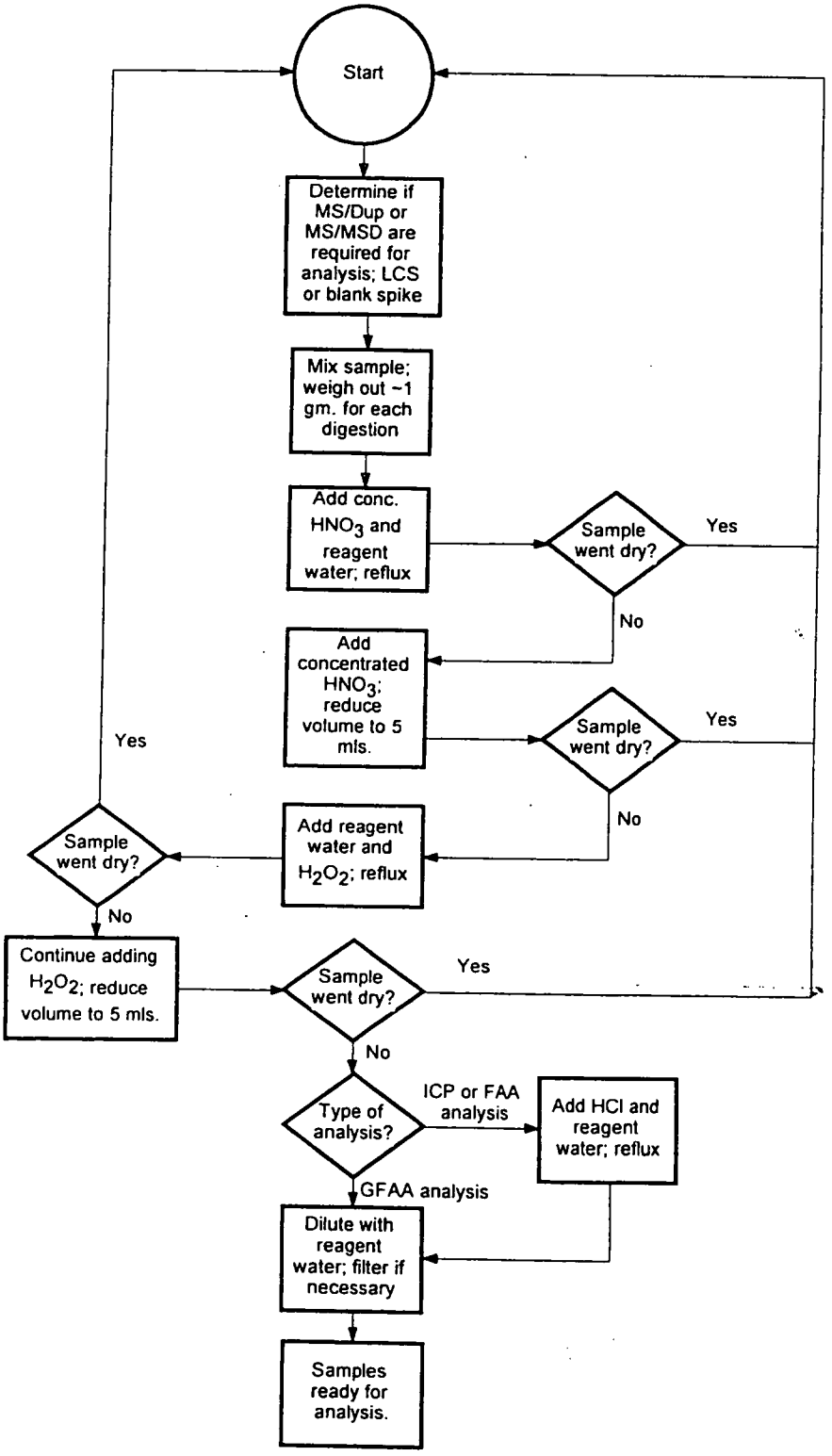
Chain of Custody	Digest Relinquished Date / Time : Analyst	Digest Accepted Analyst	Digest Disposed Date	Hot Plate Temperature, °C

Reagents Lot Number	HNO_3	HCl	H_2O_2

Color 1=Red, 2=Blue, 3=Yellow, 4=Green, 5=Orange, 6=Violet, 7=White, 8=Brown, 9=Grey, 10=Black, 11=Colorless
Clarity 1=Clear, 2=Cloudy, 3=Opaque
Texture F=Fine (powdery), M=Medium (sandy), C=Coarse (rocky) Artifacts: If "Yes", give description in the comments field.

P:\metals\benchsee\gfdig.doc

Appendix B, Flowchart for LTL-7015


Start
Determine if MS/Dup or MS/MSD are required for analysis; LCS or blank spike
Mix sample; weigh out ~1 gm. for each digestion
Add conc. HNO_3 and reagent water; reflux
Sample went dry?
Yes
No
Add concentrated HNO_3; reduce volume to 5 mls.
Sample went dry?
Yes
No
Add reagent water and H_2O_2; reflux
Sample went dry?
Yes
No
Continue adding H_2O_2; reduce volume to 5 mls.
Sample went dry?
Yes
No
Type of analysis?
ICP or FAA analysis
Add HCl and reagent water; reflux
GFAA analysis
Dilute with reagent water; filter if necessary
Samples ready for analysis.

Appendix C, Quality Control Solutions

ICP Spikes

- The CLP spike solution for ICP digest was made by diluting 10 mLs of CLPP-SPK-1™, 10 mLs of CLPP-SPK-2™, and 10 mLs of CLPP-SPK-3™ to 100 mLs with Type II water.

- Add 1000 µLs of CLP spike solution to water samples and 2000 µLs of CLP spike solution to soil samples prior to digestion.

ICP Analytes

Analyte	Concentration in Digest (ppb)	Analyte	Concentration in Digest (ppb)
Ag	50	Fe	1000
Al	2000	Mn	500
As	2000	Ni	500
Ba	2000	Pb	500
Be	50	Se	2000
Cd	50	Sb	500
Co	500	Tl	2000
Cr	200	V	500
Cu	250	Zn	500

Table based on a final volume of 100 mL for water digests, and 200 mL for soil digests.

GRAPHITE FURNACE SPIKES

- Add the analytes listed in the table below to a 100 mL volumetric flask which contains ~50 mL of Type II water and 5 mL of HNO_3.

Analyte	Volume	Stock Concentration
As	400 µL	1000 ppm
Pb	200 µL	1000 ppm
Se	100 µL	1000 ppm
Tl	500 µL	1000 ppm

- Dilute to 100 mL with Type II water. This spike solution now contains:

4.0 ppm	Arsenic
2.0 ppm	Lead
1.0 ppm	Selenium
5.0 ppm	Thallium

- Add 1000 µL of Spike Solution to water samples and 2000 µL to soil samples prior to digestion.

ICP
CLP-LOW SPIKES

- Add the analytes from the two tables below into separate 100 mL volumetric flasks which contain ~50 mL of Type II water and 5 mL of HNO_3.

ICP-1

Analyte	Volume	Stock Concentration	Spike Concentration
Sb	500 µL	1000 ppm	5 ppm
Al	5000 µL	1000 ppm	50 ppm
Ba	2000 µL	1000 ppm	20 ppm
Be	100 µL	1000 ppm	1 ppm
Cr	500 µL	1000 ppm	5 ppm
Co	1000 µL	1000 ppm	10 ppm
Cu	500 µL	1000 ppm	5 ppm
Fe	2500 µL	1000 ppm	25 ppm
Mn	500 µL	1000 ppm	5 ppm
Ni	1000 µL	1000 ppm	10 ppm
V	1000 µL	1000 ppm	10 ppm
Zn	1000 µL	1000 ppm	10 ppm

ICP-2

Analyte	Volume	Stock Concentration	Spike Concentration
Ag	500 µL	1000 ppm	5 ppm

- Add 1000 µL of ICP-1 and 1000 µL of ICP-2 spike solutions to water samples prior to digestion. For soil samples, add 2000 µL of each spike solution prior to digestion. The final digest will contain:

Analyte	Concentration in Digest	Analyte	Concentration in Digest
Sb	50 ppb	Al	500 ppb
Ba	200 ppb	Be	10 ppb

Cr	50 ppb	Co	100 ppb
Cu	50 ppb	Fe	250 ppb
Mn	50 ppb	Ni	100 ppb
V	100 ppb	Zn	100 ppb
Ag	50 ppb		

Table based on final volume of 100 mL for water digests, and 200 mL for soil digests.

GRAPHITE FURNACE
CLP-LOW SPIKES

- Add the analytes listed in the table below to a 100 mL volumetric flask which contains ~50 mL of Type II water.

Analyte	Volume	Stock Concentration	Spike Concentration
As	100 µL	1000 ppm	1000 ppb
Se	500 µL	1000 ppm	5000 ppb
Tl	500 µL	1000 ppm	5000 ppb
Pb	250 µL	1000 ppm	2500 ppb
Cd	100 µL	1000 ppm	1000 ppb

- Add 1000 µL of Spike Solution to water samples and 2000 µL to soil samples prior to digestion. The final digest will contain*:

10 ppb Arsenic
50 ppb Selenium
50 ppb Thallium
25 ppb Lead
10 ppb Cadmium

*Based on 100 mL final volume for water digests and 200 mL for soil digests.

ICP LCSW

The LCSW for the ICP digest was made by diluting 10 mLs of CLP-SPIKE and 50 mLs of LTL-6 solutions, and 2 mLs of conc. HNO_3 to 1000 mLs with type II water. A 100 mL aliquot of this solution was then digested.

ICP Analytes

Analyte	Concentration in Digest (ppb)	Analyte	Concentration in Digest (ppb)
Al	2000	Pb	500
Ba	2000	Mg	50000
Be	50	Mn	500
Cd	50	Ni	500
Ca	50000	K	50000
Cr	200	Ag	50
Co	500	Na	50000
Cu	250	V	500
Fe	1000	Zn	500
Sb	500		

Other Analytes

Analyte	Concentration in Digest (ppb)	Analyte	Concentration in Digest (ppb)
As	2000	Tl	2000
Se	2000		

Tables based on a final volume of 100 mL.

Graphite Furnace LCSW

- Add the analytes listed in the table below to a 100 mL volumetric flask which contains ~50 mL of Type II water and 2 mL of HNO_3. Dilute to volume with Type II water.

Analyte	Volume	Stock Concentration
As	500 µL	1000 ppm
Pb	500 µL	1000 ppm
Se	250 µL	1000 ppm
Tl	250 µL	1000 ppm
Cd	100 µL	1000 ppm

- Dilute to 100 mL with Type II water. This LCSW stock solution now contains:

5.0 ppm	Arsenic
5.0 ppm	Lead
2.5 ppm	Selenium
2.5 ppm	Thallium
1.0 ppm	Cadmium

The LCSW for the GFAA digest was made by diluting 10 mLs of LCSW stock solution and 20 mLs of conc. HNO_3 to 1000 mLs with type II water. A 100 mL aliquot of this solution was then digested.

Graphite Furnace Analytes

Analyte	Concentration in Digest (ppb)	Analyte	Concentration in Digest (ppb)
As	50	Tl	25
Pb	50	Cd	10
Se	25		

Tables based on a final volume of 100 mL.

PDCA
SPIKES and LCSW

The LCSW for the PDCA extraction was make by adding 10 mL SPEX ICV-1™ solution to 100 mL of Type II water prior to extraction. The spike was made by adding 1000 µL of CLP SPK solution to a 100 mL aliquot of the sample prior to extraction.

ICP Analytes

Analyte	Concentration in Digest	Analyte	Concentration in Digest
Cd	50 ppb	Mn	500 ppb
Co	500 ppb	Ni	500 ppb
Cu	250 ppb	Ag	50 ppb
Fe	1000 ppb	V	500 ppb
Zn	500 ppb		

Graphite Furnace Analytes

Analyte	Concentration in Digest	Analyte	Concentration in Digest
Pb	500 ppb	Tl	2000 ppb
Se	2000 ppb		

Tables based on a final volume of 100 mL.

APPENDIX D, PROCEDURE CHECKLIST

SW846-3050B

ICP/FAA

- [ ] 1.0-2.0g Sample
- [ ] Add Spikes
- [ ] 5 mL H_2O
- [ ] 5 mL HNO_3
- [ ] Heat 15 min
- [ ] Cool
- [ ] 5 mL HNO_3
- [ ] Heat 30 min
- [ ] Repeat (5 mL HNO_3) until no brown fumes exist
- [ ] Reduce to 5 mL or heat 2 hours
- [ ] Cool
- [ ] 2 mL H_2O
- [ ] 3 mL H_2O_2
- [ ] Heat until effervescence subsides
- [ ] Cool
- [ ] Repeat with 1 mL aliquots H_2O_2 until effervesence is minimal or 7 times
- [ ] Reduce to 5 mL or heat 2 hours
- [ ] Cool
- [ ] 10 mL HCl
- [ ] Heat 15 min
- [ ] Cool
- [ ] Final Volume 100 mL

GFAA/ICP-MS

- [ ] 1.0-2.0g Sample
- [ ] Add Spikes
- [ ] 5 mL H_2O
- [ ] 5 mL HNO_3
- [ ] Heat 15 min
- [ ] Cool
- [ ] 5 mL HNO_3
- [ ] Heat 30 min
- [ ] Repeat (5 mL HNO_3) until no brown fumes exist
- [ ] Reduce to 5 mL or heat 2 hours
- [ ] Cool
- [ ] 2 mL H_2O
- [ ] 3 mL H_2O_2
- [ ] Heat until effervescence subsides
- [ ] Cool
- [ ] Repeat with 1 mL aliquots H_2O_2 until effervesence is minimal or 7 times
- [ ] Reduce to 5 mL or heat 2 hours
- [ ] Cool
- [ ] Final Volume 100 mL

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #: LTL-7101

Title: **Operation of Jarrell-Ash Enviro 36 Simultaneous ICP**

Revision history:

Number	Date
1	01/23/96
2	01/09/97
3	2/4/98

Revised by: Katja Palmer — Date: 02/05/98
Katja Palmer, Primary ICP Operator

Approved by: Harry Romberg — Date: 2-5-98
Harry Romberg, QA Officer

Approved by: Karen J. Kotz — Date: 2/5/98
Karen Kotz, Laboratory Director

UNCONTROLLED

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1. Introduction and Scope

1.1 Purpose

1.1.1 The purpose of this SOP is to define the operation of the Jarrell-Ash Enviro 36 (JA36) ICP and any tasks associated with the operation of the instrument. Specific QC requirements and method protocols are listed in their respective SOPs.

2. Equipment List and Standards

◊ Jarrell-Ash Enviro36 simultaneous ICP with autosampler
◊ Calibration standards
◊ Standard reference materials
◊ Coolant circulator
◊ Pump and nebulizer tubing
◊ 13 x 100 mm disposable culture tubes

2.1 Standards

2.1.1 All standards are made in deionized water acidified with 5% HCl and 1% HNO_3. Each completed standard contains 50 ppm of Scandium internal standard. Expiration dates for the standards are based on the expiration date of the purchased stock standard solution. Daily working standards are made up as needed.

2.1.2 The standards used for routine ICP operation are:

◊ A Blank Standard
◊ A High Standard
◊ Mid range standards(for SW846 6010 A only)
◊ CCB (continuing calibration blank)
◊ CCV(continuing calibration verification)
◊ ICB (Initial Calibration blank)
◊ ICV (Initial calibration verification)
◊ ICSA (interference check sample A)
◊ ICSAB (interference check sample B)
◊ CRI (low detection limit sample)

2.1.3 Additionally, every quarter an IDL standard is analyzed, on three non-consecutive days. The exact concentrations of the IDL standards are listed in appendix 2. Annually, the MDL standard is digested and analyzed as part of an MDL study. The concentration of the MDL standard used for the study is documented in the MDL study file.

3. Safety precautions and Waste Disposal

3.1 Safety Precautions

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

- Sample digests are acid solutions, use routine acid handling care.
- Concentrated metals standards should be handled with care so that none are absorbed through the skin.

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

3.1.3 Do not look directly at the torch without the polarizing shield as the UV rays emitted will cause eye damage.

3.1.4 Do not try to operate the instrument with the front door of the torch compartment open by bypassing the interlock.

3.1.5 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.

3.1.6 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.

- Do not attempt to change the power amplifier tube as extremely high voltage may cause electrical shock. Call service engineer to perform this task.
- Do not remove any panels of the RF generator as exposure to microwaves will occur.

3.2 Waste Disposal

3.2.1 Waste is collected in the carboy under the torch compartment. Empty as necessary. Always leave about six-inches of waste in the bottom.

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 Calibration Procedure

4.1.1 A SPEX multi-element standard and a Blank standard are used to calibrate the instrument. If running in the autosampler mode, the standards are included at the beginning of the autosampler table.

4.1.2 In the manual mode, select **Operation** from the main menu then select **Analysis.**

4.1.3 Enter the name of the method used or accept the default method. Press F3 for standardization.

4.1.4 Use the arrow keys to highlight the name of the standard. Aspirate the standard and press "F1" (run). Repeat until all standards have been run. Press "F9" twice to return to the analysis menu.

4.2 Quality Control and Limit Check Tables

Note: These tables are method specific (SW846, 200.7 and CLP) and must be applied appropriately.

4.2.1 Quality control check tables are used with QC samples. They contain the true values of the ICVs, the acceptable range of the results according to the method being run, and the units of comparison: percent or absolute. QC Check tables are only used when a sample is defined as a QC sample.

4.2.2 Limit check tables are used with analytical samples and with blank samples (i.e. ICBs and CCBs). The control limits for blanks are dependent on the method being run. They are generally defined as being the absolute value of the result. They should not exceed the reporting limit. For analytical samples their upper range is set as the linear range of the instrument or the level of the high standard, depending on the specifications of the method being run. The lower limit is set at the negative reporting limit, again method specific.

4.2.3 Method information and the QC and Limit tables for each method can be found in **Appendix 3.**

5. Operation procedures

5.1 Startup

- The argon tanks, located in the warehouse, are connected to a delivery manifold that will allow a continuous flow of argon as long as there is 90 psi available in a tank. The line-in pressure regulator is located behind the instrument and is to be adjusted to 50 psi.

- Check pump tubing to see if it is flat or crimped. Change if necessary. Check all connections to see if any blockage exists and if clogs are found, remove them by disconnecting the tubing and flushing with DIW or by using a cleaning wire.

- Turn on control and line breakers on the RF generator.

- Turn on the computer power strip and the power strip attached to the autosampler cart.

- Turn on the torch, auxiliary, and sample argon toggles on the front of the instrument. Let the argon purge through the system for about 3 minutes.

- Settings: torch=18
  aux=0.5
  sample=0.65.

- At the computer select OPERATION, ANALYSIS, accept the default method, and at the command line type **iara** <ENTER>.

- Stretch the rinsate pump tubing into place, set the pressure plate, and turn on the peristaltic pump.

- Prior to igniting the torch aspirate rinse water into the nebulizer. Observe the spray chamber to ensure the aspirated solution is being nebulized.

- Turn off the sample argon toggle to halt gas flow into the nebulizer.

- Press the red RF ON button, then turn the power knob until the forward power reads about 0.5 on the meter.

- Press the ignite button to light the torch. If the torch does not light the first time try again. If it still does not light, adjust the forward power slightly higher until the torch lights while pressing the ignite button. The torch is lit when white streams appear at the top of the torch.

- Slowly increase power until a plasma forms (white "flame"). This will be accompanied by "popping" sounds as the plasma begins to form.

- If the torch appears yellow-orange during this process, depress the blue RF OFF button immediately and turn the power knob back to the off position. This yellow-orange color means that the torch is overheating. See the manufacturers manual, page 14, for corrective action.

- Adjust the forward power to 1.1 KW.

- Switch the automatic power toggle from manual to automatic.

- Slowly turn the power knob fully clockwise.

5.2 Automatic Profiling

- Select OPERATION from the main menu.

- Select ANALYSIS from the menu.

- Accept the default method.

- Set the 5 ppm Mn profiling solution on the autosampler tray at rack #2 position 75 (the white dotted circle). At the command line of the software type **iasrn2srt70ma75** <ENTER>. Translation: Initialize Autosampler, Set Rack Number 2, Set Rack Type 70, Move Autosampler to position #75.

- Select F5 (Profile) from the function keys.

- Select F3 (Automatic) from the function keys.

- Type in a 40 second flush time.

- Press F1 (Run) from the function keys.

- If the peak is between 0.1 and -0.1, the instrument is profiled. If the peak is outside of this range a manual profile is required.

5.3 Manual Profiling

- With the Mn standard still being aspirated, select F1 (manual profile) from the Profile menu.
- Moving the micrometer screw, maximize the reading on the profile meter.
- Move the micrometer screw clockwise until the profile meter reads less than 70% of the maximum. Record the value of the micrometer screw.
- Continue moving the micrometer screw counterclockwise, past the maximum reading on the profile meter, until the profile meter once again reads less than 70% of the maximum. Record the value of the micrometer screw.
- Determine the average of the two values.
- Set the micrometer at the average value by moving the screw in a counterclockwise direction.
- Repeat the auto-profile to check the peak position.

5.4 Analytical Operation

5.4.1 All analytical solutions aspirated into the ICP must contain 50 ppm Sc. 50 µL of 10000 ppm scandium is added to a 10 ml aliquot of sample. If samples are to be run manually aspirate right out of the tube. If they are to be included in an autosampler run, pour the sample into a culture tube and load the tube into the appropriate spot in the autosampler tray.

5.4.2 If samples are to be run in the autosampler mode it is necessary to set up the autosampler table. From the menu choose **Autosampler setup.** Select the table name. Press "F1"(edit autosampler table). Type in the sequence of samples.

5.4.3 To analyze samples in the manual mode it is necessary to be in the **Analysis** menu. After the instrument has been calibrated, press "F2". Fill in the requested information. Aspirate the ICV. Press "F1"(run). For the ICB, use "F4"(Blank sample). For analytical samples use "F1"(analyze). Repeat for additional reference samples and then for "real" samples.

5.4.4 To analyze samples in the autosampler mode it is also necessary to be in the **Analysis** menu. Enter the desired method or accept the default Method. Calibration will be done by the autosampler table. Press "F9"(Autosampler). Type in the name of the autosampler table and press the "ENTER" key. Verify that the standards and the samples are in the correct positions. Press " F1"(start).

5.4.5 For more details on creating Tables and Methods refer to the Operators Manual for the ICP.

5.5 Shutdown Procedure

5.5.1 Turn the power knob on the RF generator off (left) and press the red reset button when the alarm sounds. Press the blue RF OFF button. Then flip the toggle switch for the automatic power control to the manual position.

5.5.2 The control and line breaker switches only need to be turned off for weekends or if the instrument is not going to be used the next day. This should be done after the RF power has been off for at least 5 minutes.

5.5.3 Turn off the power strip on the autosampler cart.

5.5.4 Release the pressure plates on the peristaltic pump and unhook the pump tubing.

5.5.5 Turn off the torch, auxiliary, and sample argon toggles on the front of the instrument.

5.5.6 If the computer will not be used, turn off the master switch on the computer power strip.

6. Reports

6.1 Results

6.1.1 All Results are organized into Data packets. The format of each packet is dependent on the method and protocol required for that specific job. Details for method specific reports can be found in the method SOP.

6.2 Quality Control Reports

6.2.1 All results for quality control tests are entered into the lab data base using the Quality Control database QC_DB. Printouts of all data entered must be included in the data

packet. The routine minimum is a method blank report, and an MS/MSD or MS/duplicate report. Many analyses will also require an SRM (LCSS), blank spike (LCSW) or other report. The recovery values for Ag,Al,Cr,Cd and B in the LCSS and LCSW are plotted on control charts.

6.2.2 Method specific QC requirements are listed in the Method SOP.

7. Quarterly and Yearly Procedures

7.1 Instrument Detection Limits (IDLs)

7.1.1 For complete details on performing IDL studies refer to Laucks SOP LTL-1011. In brief, IDLs for each analyte are determined quarterly by analyzing a "sample" seven consecutive times. This is done on three non-consecutive days. Calculate the standard deviation of the seven results on a given day. Add the standard deviations from the three days and the result is the IDL. The IDL solution is at a concentration of 3-5X the expected IDL. The IDL standard is not digested. The exact concentrations for the IDL standard are listed in **Appendix 2** of the SOP.

7.2 Method Detection Limits (MDLs)

7.2.1 For complete details on performing IDL studies refer to Laucks SOP LTL-1011 . In brief, MDLs for each analyte are determined yearly by analyzing seven replicates of a digested solution. MDLs are run according to the Laucks SOP on MDL determinations using the appropriate method. The MDL solution concentration is documented in the MDL study file.

7.3 Linear Range

7.3.1 CLP and EPA 200.7 - 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 linear range. Linear range must be verified quarterly. A list of the ICP linear ranges can be found in **Appendix 2** of this document.

7.3.2 SW846 6010B - The upper limit of the linear dynamic range must be established by running a multi-point calibration using 4 standards and then running an upper range standard. The upper range limit should be an observed signal with no more than a 10%

variation from the true value of the upper range standard. Linear range must be checked every six months.

7.4 Interelement Correction Factors (IECs)

7.4.1 Interelement correction factors are determined by analyzing single element, high concentration samples. The apparent concentration of an analyte divided by the concentration of an interfering analyte run at 100 ppm, gives the required correction for that analyte/interfering element combination. This must be verified and updated for all analytes and all interfering elements every six months.

Appendix 1

Concentration of analytes in standard solutions in mg/L for CLP/200.7 and SW846 6010B

ELEMENT	ICAL STD	ICV	CCV	ICSA	ICSAB	CRI
Ag	1.0	0.6	0.5	-	1	0.02
Al	20.	12	10	500	500	-
As	1.0	2.6	2.5	-	1	0.02
Ba	20.	12	10	-	0.5	-
Be	0.5	0.3	0.25	-	0.5	0.01
Ca	50.	300	25	500	500	-
Cd	0.5	0.3	0.25	-	1	0.01
Co	5.0	3	2.5	-	0.5	0.10
Cr	1.0	0.6	0.5	-	0.5	0.02
Cu	2.5	1.5	1.25	200	0.5	0.05
Fe	10.	6	5	-	200	-
Hg	2.0	2	1	-	-	-
K	50.	300	25	500	-	-
Mg	50.	300	25	-	500	-
Mn	1.5	0.9	0.75	-	0.5	0.03
Na	50.	300	25	-	-	-
Ni	4.0	2.4	2	-	1	0.08
Pb	0.5	2.3	1	-	1	0.056
Sb	6.0	3.6	3	-	-	0.12
Se	0.5	2.3	2.5	-	1	0.01
Tl	1.0	0.6	5	-	1	0.02
V	5.0	3	2.5	-	0.5	0.10
Zn	2.0	1.2	1	-	1	0.04
Mo	5.0	10	2.5	-	-	-
B	5.0	10	2.5	-	-	-

* ICV and CCV have different stock solutions

Appendix 1. cont.

Concentration of analytes for SW846 6010A calibration standards in mg/l

ELEMENT	Std 1 (low std)	Std 2 (mid std)	Std 3 (mid-hi std)	Std 4 (high std)
Ag	0.02	0.2	1.0	2.0
Al	1.0	10.	50.	100.
As	0.05	0.5	2.5	5.0
Ba	0.2	1.0	5.0	10.
Be	0.01	0.1	0.5	1.0
Ca	4.0	40	200.	400.
Cd	0.02	0.2	1.0	2.0
Co	0.05	0.5	2.5	5.0
Cr	0.05	0.5	2.5	5.0
Cu	0.05	5.0	25.	50.
Fe	2.0	20.	100.	200
Hg	0.05	0.5	2.5	5.0
K	3.0	30.	150.	300
Mg	2.0	20.	100.	200.
Mn	0.1	1.0	5.0	10.
Na	2.5	25	125.	250.
Ni	0.1	1.0	5.0	10.
Pb	0.1	5.0	25.	50.
Sb	0.05	0.5	2.5	5.0
Se	0.05	0.5	2.5	5.0
Tl	0.1	1.0	5.0	10.
V	0.1	1.0	5.0	10.
Zn	0.1	5.0	25.	50
Mo	0.2	1.0	5.0	10.

* SW846 H.std=ICV

Appendix 2

IDL solutions and linear range for CLP/200.7

ELEMENT	IDL solution T.V. (mg/L)	linear range (mg/L)
Ag	0.01	200.
Al	0.05	600.
As	0.04	300.
Ba	0.01	100
Be	0.002	10.
Ca	0.05	600
Cd	0.005	100
Co	0.01	200
Cr	0.02	200
Cu	0.01	200
Fe	0.05	600
Hg	0.04	-
K	0.45	1000
Mg	0.05	600
Mn	0.02	100
Na	0.05	600
Ni	0.01	200
Pb	0.04	200
Sb	0.02	200
Se	0.05	200
Tl	0.05	10
V	0.04	200
Zn	0.002	200
Mo	0.01	-
B	0.2	-

Appendix 3

Method information, QC and limit check tables

Method: LAUCKIEC

Applicable to CLP/200.7

METHOD INFORMATION

Sample Introduction Device: Normal
Calibration Mode: Concentration

Default Setup:

Number of Repeats :	4	Auto-store Analysis Data?	Yes
Flush Time (sec) :	60.0	Auto-store Stdzn Data?	No
Auto-Increment Sample Names?	No	Store Individual Repeats?	Yes
		Auto-print Analysis Data?	Yes
		Auto-print Stdzn Report?	None
		Condensed Print Format?	No

Default File Names:

Analysis Data File	: MIKEDAT	Autosampler Table	: AUTO
		Sample Limits Table	: LINCHK
Calibration Data File	: DEFAULT	Blank Limits Table	: MGLBLK
Calibration Stds Table	: DEFAULT	QC Check Table	SPEXCCVM

Limit Check Table LINCHK
limit values in ppm

ELEMENT	High limit	Low limit
Ag*	200.	-.01
Al*	600.	-.2
As*	300.	-.3
Ba*	200.	-.1
Be*	100.	-.005
Ca*	600.	-1.
Cd	100.	-.005
Co	200.	-.05
Cr	200.	-.01
Cu	200.	-.025
Fe	600.	-.1
Mg	600.	-1.
Mn	100.	-.015
Na	600.	-1
Ni	200.	-.04
Pb	200.	-.1
Sb	200.	-.06
Se	200.	-.3
Tl	10.	-.5
V	200.	-.05
Zn	200.	-.02
K	1000	-1
B	100.	-.2
Mo	100.	-.01

Limit Check Table MGLBLK
limit values in ppm

Element	High limit	Low limit
Ag*	.01	-.01
Al*	.2	-.2
As*	.2	-.2
Ba*	.2	-.2
Be*	.005	-.005
Ca*	1.	-1.
Cd*	.005	-005
Co*	.05	-.05
Cr*	.01	-.01
Cu*	.025	-.025
Fe*	.1	-.1
Mg*	1.	-1.
Mn*	.015	-.015
Na*	1.	-1.
Ni*	.04	-.04
Pb*	.05	-.05
Sb*	.06	-.06
Se*	.2	-.2
Tl*	.5	-.5
V*	.05	-.05
Zn*	.02	-.02
K*	1.	-1.

QC Check Table SPEXICVM and BMOICVM
limit values in ppm

Element	Value	% Range
Ag*	.6	10
Al*	12.	10
As*	2.6	10
Ba*	12.	10
Be*	.3	10
Ca*	300.	10
Cd*	.3	10
Co*	3	10
Cr*	.6	10
Cu*	1.5	10
Fe*	6.	10
Mg*	300.	10
Mn*	.9	10
Ni*	2.4	10
Pb*	2.3	10
Sb*	3.6	10
Se*	2.3	10
Tl*	.6	10
V*	3.	10
Zn*	1.2	10
K*	300.	10
B	10	10
Mo	10	10

QC Check Table SPEXCCVM
limit values in ppm

Element	Value	% Range
Ag*	.5	10
Al*	10	10
As*	2.5	10
Ba*	10	10
Be*	.25	10
Ca*	25	10
Cd*	.25	10
Co*	2.5	10
Cr*	.5	10
Cu*	1.25	10
Fe*	5	10
Mg*	25	10
Mn*	.75	10
Na*	25	10
Ni*	2	10
Pb*	1	10
Sb*	3	10
Se*	2.5	10
Tl*	5	10
V*	2.5	10
Zn*	1	10
K*	25	10
Hg*	1	10
B*	2.5	10
Mo*	2.5	10

* QC Check Table SPEXCCVM valid for CCVs in all current Methods

Method: SW846

Applicable to SW846 Soils

METHOD INFORMATION

Sample Introduction Device: Normal
Calibration Mode: Concentration

Default Setup:

Number of Repeats : 4
Flush Time (sec) : 60.0
Auto-Increment Sample Names? No

Auto-store Analysis Data? Yes
Auto-store Stdzn Data? No
Store Individual Repeats? Yes
Auto-print Analysis Data? Yes
Auto-print Stdzn Report : None
Condensed Print Format? No

Default File Names:

Analysis Data File : MIKEDAT
Calibration Data File : DEFAULT
Calibration Stds Table: DEFAULT

Autosampler Table : AUTO
Sample Limits Table : HCALSTD
Blank Limits Table : MDL2X
QC Check Table : SPEXCCVM

Limit Check Table HICALSTD
limit values in ppm

Element	High limit	Low Limit
Ag*	2	-.0055
Al*	100	-.221
As*	5	-.057
Ba*	10	-.038
Be*	1	-.003
Ca*	400	-1.008
Cd*	2	-0063
Co*	5	-.0129
Cr*	5	-.0199
Cu*	50	-.0177
Fe*	200	-.4988
Mg*	200	-.5198
Mn*	10	-.0247
Na*	250	-.5528
Ni*	10	-.0365
Pb*	50	-.059
Sb*	5	-.033
Se*	5	-.1005
Tl*	10	-.0668
V*	10	-.0255
Zn*	50	-.036
K*	300	-.5472
MO*	10	-0783

* HCALSTD Table low limit based on 2X current soil MDL and subject to change annually

Limit Check Table MDL2X
limit values in ppm

Element	High Limit	Low Limit
Ag*	.0055	-.0055
Al*	.221	-.221
As*	.057	-.057
Ba*	.038	-.038
Be*	.003	-.003
Ca*	1.008	-1.008
Cd*	.0063	-.0063
Co*	.0129	-.0129
Cr	.0199	-.0199
Cu	.0177	-.0177
Fe*	.4988	-.4988
Mg*	.5198	-.5198
Mn*	.0247	-.0247
Na*	.5528	-.5528
Ni*	.0365	-.0365
Pb*	.059	-.059
Sb*	.033	-.033
Se*	.1005	-.1005
Tl*	.0668	-.0668
V*	.0255	-.0255
Zn*	.036	-.036
K*	.5472	-.5472
Mo*	.0783	-.0783

* Blank Limits based on 2X soil MDL and therefore subject to annual variations

QC Check Table HSTDICVM
limit values in ppm

Element	Value	% Range
Ag*	2	5
As*	5	5
Ba*	10	5
Ca*	400	5
Be*	1	5
Al*	100	5
Cd*	2	5
Co*	5	5
Cr*	5	5
Cu*	50	5
Fe*	200	5
Hg*	5	5
K*	300	5
Mg*	200	5
Mn*	10	5
Na*	250	5
Ni*	10	5
Pb*	50	5
Sb*	5	5
Se*	5	5
Tl*	10	5
V	10	5
Zn*	50	5
Mo*	10	5

Method: SW846W

Applicable to SW846 Waters

METHOD INFORMATION

Sample Introduction Device: Normal
Calibration Mode: Concentration

Default Setup:

Number of Repeats 4
Flush Time (sec) 60.0
Auto-Increment Sample Names? No

Auto-store Analysis Data? Yes
Auto-store Stdzn Data? No
Store Individual Repeats? Yes
Auto-print Analysis Data? Yes
Auto-print Stdzn Report : None
Condensed Print Format? No

Default File Names:

Analysis Data File: MIKEDAT
Calibration Data File: DEFAULT
Calibration Stds Table: DEFAULT
Calibration Stds Table: DEFAULT

Autosampler Table AUTO
Sample Limits Table HCALSTDW
Blank Limits Table WMDL2X
QC Check Table SPEXCCVM

Limit Check Table HCALSTDW
limit values in ppm

Element	High Limit	Low Limit
Ag*	2	-.0082
Al*	100	-.1864
As*	5	-.0528
Ba*	10	-.0119
Be*	1	-.001
Ca*	400	-.346
Cd*	2	-.0213
Co*	5	-.0057
Cr *	5	-.0157
Cu*	50	-.0051
Fe*	200	-.163
Mg*	200	-.253
Mn*	10	-.008
Na*	250	-.1975
Ni*	10	-.0086
Pb*	50	-.04
Sb*	5	-.0154
Se*	5	-.0698
Tl	10	-.071
V*	10	-.0051
Zn*	50	-.0147
K*	300	-.288
Mo*	10	-.0233

* Low sample limit bases on 2X Water MDL. Subject to change annually.

Limit Check Table WMDL2X
limit values in ppm

Element	High Limit	Low Limit
Ag*	.0082	-.0082
Al*	.1864	-.1864
As*	.0528	-.0528
Ba*	.0119	-.0119
Be*	.0009	-.0009
Ca*	.346	-.346
Cd*	.0213	-.0213
Co*	.0057	-.0057
Cr*	.0157	-.0157
Cu*	.0051	.0051
Fe*	.163	-.163
Mg*	.253	-.253
Mn*	.008	-.008
Na*	.1975	-.1975
Ni*	.0086	.0086
Pb*	.04	-.04
Sb*	.0154	-.0154
Se*	.0698	-.0698
Tl*	.071	-.071
V*	.0051	-.0051
Zn*	.0147	-.0147
K*	.288	-.288
Mo*	.0233	-.0233

* Blank limits bases on current Water MDLs. Subject to annual variation.

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-7105

Title: Method Protocols for the Analysis of Metals by ICP Using SW 846 6010B

Revision history:

Number	Date
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1	03/24/00

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No. 20 Assigned to: Tetra

Revised by: _____ Date: 03/24/2000
Paul Swift, Metal Supervisor

Approved by: _____ Date: 3-24-2000
Harry Romberg, Quality Assurance Officer

Approved by: _____ Date: 03-24-2000
Kathy Kreps, Laboratory Director

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Introduction and Scope

1.1 Scope

This method is to be used as a supplement to the instrumental SOP in order to follow the method requirements of SW 846 6010B for ICP analysis. Operating parameters are to be followed in the individual instrument SOP.

The analyst should become familiar with SW 846 protocols prior to using this SOP.

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

1.2 Sample Collection, Sample Storage, Holding Times

Samples are to be collected in either glass or plastic containers. Water samples are to be preserved to a pH < 2 using nitric acid. A one liter sample bottle is sufficient volume for analysis. Soil samples do not require preservation but need to be stored at 4° C. At least 200 grams of sample should be collected. The holding time for ICP metals is 6 months. See Appendix III for Sample Handling and Preservation Table.

1.3 Definition of Terms

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

ICV - Initial Calibration verification - This is a standard run immediately following the initial calibration. The ICV is made from an independent source. Agreement within 10% and a relative standard deviation less than 5% RSD from replicate (minimum of two) integrations, is required.

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

CCV - Continuing calibration verification. - This is a standard analyzed after every 10 samples during the analysis sequence to determine whether the instrument or system has remained in calibration. Agreement within 10% and less than 5% RSD from replicate (minimum of two) integrations, is required.

CCB - Continuing Calibration Blank - This is a blank that is used to determine if there is carry-over between sample injections. A CCB succeeds every CCV, every 10 samples.

CRDL - Contract Required Detection Limit - The minimum level of detection at ±20% analyte recovery. This limit represents the analyte concentrations below which data must be evaluated and qualified as estimated concentrations. The commercial CRDL solution may be used as a check standard, but must be fortified with As, Pb, and Se to a final concentration before analysis of 10ug/L.

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

IEC - Interelement Correction Factors - IECs are experimentally-determined constants used by the calibration modelling software in estimating the extent of intereference caused by IEC elements on the other analyzed elements. This list is to include the nine interferents listed in SW846 Method 6010B - Al, Ca, Cr, Cu, Fe, Mg, Mn, Tl, and V.

ICSA - Interference Check Solution A - This solution is prepared to contain known concentrations of interfering elements that will provide an adequate test of the correction factors.

ICSAB - Interference Check Solution AB - This solution is prepared by spiking the ICSA with known quantities of analyte. Adequate recovery of the analytes within this interfering matrix indicates proper application of the correction factors.

IDL - Instrument detection limit - The lowest concentration of a target analyte that is detectable. The IDL is three times the average standard deviation of seven replicates at 2 to 5 times the estimated IDL over three non consecutive days. Used as a starting point for selecting MDL study spiking levels. IDL should be determined quarterly.

Method Blank Sample / Preparation Blanks Sample- A sample containing only reagent and representative matrix (DIW or blank solid material, as appropriate). This sample is to be carried through the entire preparation procedure and analyzed as a regular sample. The level of analytes in this sample must not exceed one-half the concentrations requested as reporting limits for the project, or twice the MDL, whichever is lower. Corrective action will be either qualification of data associated with that blank sample or repreparation/reanalysis of the associated samples.

MS - Matrix spike. The matrix spike sample is to be prepared such that the amounts of analyte added are near the action levels of concern for the project, but above 20x the MDLs. Preparation of the matrix spike sample is described in the appropriate SOP.

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

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

Post digestion spike - The sample selected for QC analysis will be post-spiked and analyzed in the event that the matrix spike sample exceeds recovery criteria. The final spike level should be identical to the predigestion spike, near the action levels of concern for the project but above 20x the MDL.

QC period - Quality control period - An analysis sequence initiated by the analysis of one or more standards, followed by samples, and terminated with a standard and blank analysis.

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

Sequence - A set of sample digests and standard solutions introduced into an instrument in a chronologically continuous group.

2. Equipment List and Standards

2.1 Instrumentation:

Thermo Jarrell Ash Enviro 36 simultaneous ICP, Thermo Jerrall Ash ICAP 61E Trace Analyzer or equivalent.

2.2 Standards
SW 846 Method 6010B requires the use of one standard and a blank. See the instrument SOP for standards and their preparation.

3. Safety precautions and Waste Disposal

See instrument SOP for detailed safety precautions and waste disposal.

Safety

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

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

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

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

Caution should be used when handling acidic digestates.

3.1 Waste Disposal

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

4. Calibration and Quality Control

4.1 Method Detection Limit Study

Prior to the analysis of any samples, it is necessary to establish method detection limits. This procedure is fully described in the Laucks SOP on performing MDL studies. Briefly, it involves the analysis of 7 replicate samples spiked at a concentration approximately 3 to 5 times the estimated method detection limit. A Student's T-test is then applied to these measured values to calculate the MDL.

4.2 Linear range study

The upper limit of the linear dynamic range must be established by running a multi-point calibration using 4 standards and then running an upper range standard. The upper range

limit should be an observed signal with no more than a 10% variation from the true value of the upper range standard. All samples with elements exceeding the level of the upper range standard must be diluted.

4.3 Initial Calibration and Preliminary Calibration Check Standards

Analyze standard solutions using a minimum of a calibration blank and one standard. The calibration curve must be verified by running an Initial Calibration Standard (ICV) and obtaining agreement within 10% of the expected concentration and a 5% RSD for replicate integrations.

One check standard at the CRDL is to be analyzed, and the recovery criteria shall be ±20%.

One check standard at or below the linear range for each analyte may be analyzed in order to confirm the linearity of the calibration curve between the single-point high standard and the estimated linear range maxima. Recovery of 90-110% allows the use of the calibration curve in this region to quantify sample results.

Criteria and Corrective Action:

Calibration - Since a linear regression is not possible when using a two point calibration on the Enviro 36, the standard curve is validated by evaluating the ICV and the subsequent CCVs. If the corresponding control limits for the ICV and CCV are exceeded, then the sample analysis must be discontinued, the cause determined and the instrument recalibrated. All samples following the last acceptable ICV, CCV must be reanalyzed.

Linear range standard - If this standard is not included in the analysis then any data acquired with concentration values above the high standard but within the linear range of the instrument must be qualified as estimated quantities. Preferably, the samples would be diluted and reanalyzed

CRDL - If the stated criterion cannot be met then the reporting limit will be adjusted such that this analysis will be in control. This may involve adjusting the CRDL concentration for individual analytes which failed to meet acceptance followed by reanalysis.

4.4 Initial Calibration Standard ICV:

Immediately after the calibration curve, an ICV is analyzed.

The calculated concentration of the standard must be within 10% of the expected value and the RSD must be less than 5% for replicate integrations.

Corrective Action:

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

4.5 Initial Calibration Blank

After the analysis of the high standard, an instrument blank (ICB) is analyzed. The levels of target analytes in the ICB should not exceed three times the instrument detection limit.

Corrective Action:

If the ICB analyte levels exceed 3 times the IDL, repeat the analysis two more times and average the results. If the average is not within three standard deviations of the background mean, the system is out of control. The source of contamination must be identified and corrected before proceeding with the analysis. If the blank is less than 1/10 the concentration of the action level of interest, and no sample is within 10% of the action limit, analyses need not be rerun and recalibration need not be performed before continuation of the run.

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

A continuing calibration verification standard is analyzed after every 10 samples. Immediately following the CCV, a blank solution is analyzed. In addition, this standard and blank must be the last samples analyzed in the run. The CCV must also be prepared from an independent source.

The CCV must fall within ± 10% of the true value and the RSD must be less than 5% for replicate integrations.

The levels of target analytes in the CCB should not exceed 3 times the IDL.

Corrective action:

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

If the CCB analyte levels exceed 3 times the IDL, repeat the analysis two more times and average the results. If the average is not within three standard deviations of the background

mean, terminate the analysis, correct the problem, recalibrate, and reanalyze the previous 10 samples. If the blank is less than 1/10 the concentration of the action level of interest, and no sample is within ten percent of the action limit, analyses need not be rerun and recalibration need not be preformed before continuation of the run.

4.7 Interference Check Solutions A (ICSA) and AB (ICSAB)

(ICSA):

At the beginning and at the end of each run, an interference check solution A is analyzed. This solution contains interefering elements only. All other elements are not present in the solution. All elements not present should show a recovery of zero, or ± the CRDL.

Corrective Action:
If the analytes do not recover within the specified control limits, then the system is out of control. The problem needs to be identified and corrected prior to beginning another run.

(ICSAB):

At the beginning and end of each analytical sequence an ICSAB must be analyzed. Analytes must recover between 80-120%.

Corrective Action:

If the analytes do not recover within the specified control limits, then the system is out of control. The problem needs to be identified and corrected prior to beginning another analysis.

4.8 Method Blanks

Method blanks are used to verify contamination free reagents and apparatus. They are prepared with every set of samples prepared at the same time or at least one blank every 20 samples, which ever is more frequent. Any analyte response above the MDL is reported. For a method blank to be acceptable for use with the accompanying samples, the concentration of the blank of any analyte of concern should not be higher than the highest of either:

(1) One-half of the reporting limit, which is approximately twice the value of the MDL, 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 was detected in the blank but not in any of the associated samples then sample group may not require re-analysis. In any case, if re-preparation and re-analysis is not being undertaken, the analyst must first discuss the issue with the Quality Control Officer. It is the laboratory's responsibility to ensure that method interference caused by contaminants in acids, solvents, reagents, glassware, and other sample processing hardware leading to discrete artifacts and/or elevated baselines in the analytical run be minimized. In the extreme case of chronic contamination, blanks may have to be analyzed from each stage of the sample processing to determine the contamination source so it can be eliminated. In all cases where blank contamination exceeds the control limit, a narrative comment must be made which documents the corrective actions taken.

4.9 Laboratory Control Sample

The LCS is made from an independent source of the same matrix (soil or water) and is carried through the entire digestion procedure. The LCSW is to be spiked at a level near the action levels of concern for the project but above 20x the MDL. The SDG Routing Form will indicate appropriate spike level. An LCS is performed with each digestion batch. At a minimum, LCSW(water) control limit are 80% to 120%. Control limits for the LCSW will be empirically determined and must be within the method specified control limit noted aboved.

LCSS (soil) control limits are supplied by the manufacturer. LCSS control limits are not derived by the laboratory due to the small number of data points available from each lot of certified material.

Corrective Action

If the LCS is not within the required control limits but the matrix spike is in control, corrective action may not be required unless otherwise specified. If both LCS and matrix spikes exceed limits, particularly if the recoveries are low <u>or</u> there are reportable quantities of the affected target analytes, then a redigestion will occur for the affected analytes. Other extenuating circumstances may exist that allow for exceptions without redigestion and reanalysis but they must first be approved by QA.

4.10 Matrix Spike

Unless specific samples are specified for use as matrix Spike Samples, 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. Spike samples will be identified on the SDG Routing Forms as

required. The analyst should attempt to avoid selecting samples which are identified by the client as blanks.

The matrix spike sample is to be prepared such that the amounts of analyte added are near the action levels of concern for the project, but above 20x the MDLs. Preparation of the matrix spike sample is described in the appropriate SOP.

As the purpose of the matrix spike is to test the system under "typical" conditions, the analyst may also avoid selecting the most difficult sample of the batch for spiking. It is not always required that a matrix spike analysis be performed with each preparation/analysis-batch, however, the minimum frequency for MS analysis is 1 each per 20 samples per matrix. This will be best accomplished by running one with every batch for many analyses. This matrix spike sample is used to evaluate the matrix effect of the sample upon recovery of the analytes. The recovery of spike analytes is calculated as follows:

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

where:

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

For ICP, control limits for spike recoveries will be 75-125% unless otherwise specified in the project specific QAPP. In-house control limits are based on historical performance. The recovery criteria are detailed in the QC Database QC_DB and will change from time to time.

Corrective Action:

Samples with spike recoveries outside control limits will be reviewed for possible corrective action. Corrective action will first involve recalculation, followed by possible re-preparation, and/or reanalysis. This process should also look at the recovery of matrix spiking compounds from the SRM and/or blank spike analysis.

In some cases a Post Digestion Spike is required when matrix interference is suspected. The sample selected for QC analysis will be post-spiked and analyzed in the event that the matrix spike sample exceeds recovery criteria. The final spike level should be identical to the predigestion spike, near the action levels of concern for the project but above 20x the MDL. The same 75-125% recovery criteria exist for this spike as for the matrix spike or a matrix effect is expected. In all cases a narrative explanation of the condition is required to detail

the corrective actions taken. Data reported in validatable packages will be flagged with an "N" indicating the out-of-control event..

4.11 Matrix Spike Duplicate/Sample Duplicate

Method QC consists of MS/MSD. A duplicate maybe be performed instead of a MSD. Other types of QC can performed at the client's request.

Criteria

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

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

where:
SS = concentration in spiked sample
SSD = concentration in matrix spiked duplicate sample

For sample concentractions greater than ten times the IDL, control limits for RPD of duplicates will be ±20% unless otherwise specified in the project specific QAPP. For sample concentrations less than ten times the IDL, control limits for the difference will be ± the reporting limit. In-house control limits are based on historical performance. The RPD control limits are detailed in the current QC Database QC_DB and will change from time to time.

Corrective Action:

If a trend in out of control RPD values is observed, the methods used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable reproducibility. Generally, if recoveries are in control and no analyte of interest was detected in any of the samples, no immediate action will be taken on that sample set. If integrity of reported sample values is in doubt, re-analysis may be called for. Corrective actions should be discussed with the Quality Control Officer. In a validatable package, data associated with an out-of-control RPD will be flagged with an "*".

4.12 Serial Dilution

A five-fold dilution is performed on the QC sample in each analytical batch. The difference between the initial value and the serial dilution should agree within 10%. If the difference is greater than 10% on analytes that exhibit a level 50 times greater than the MDL, then results for those analytes will be flagged with an "E".

Corrective Action:

No corrective action is necessary other than appropriately flagging the data.

5. Operation procedures

- Calibrate the instrument with a blank and one standard.
- Immediately following the calibration, analyze the ICV followed by an ICB. Concentration values obtained for the ICV should not deviate from the actual values by more than 10% and less than 5% RSD for replicate integrations.
- Analyze both the CRDL standard and the linear range standard. The CRDL recoveries should not exceed ±20% and the linear range standard should not deviate from the actual values by more than 10% and less than 5% RSD for replicate integrations.
- Flush the system with the calibration blank solution for at least 1 minute before the analysis of each sample.
- Analyze a CCV and CCB every 10 samples. The CCV must agree within 10% of its expected value and less than 5% RSD for replicate integrations. The results of the calibration blanks are to be within 3 times the IDL. If not, repeat the analysis two or more times and average the results. If the average is not within 3 times the IDL, terminate the analysis, correct the problem, recalibrate, and reanalyze the previous 10 samples.

5.1 Analysis Sequence

See Appendix IV for Analytical Sequence

5.2 Instrumental Conditions

See instrument SOP for operating procedures.

5.3 Analytical Operation

See instrument SOP for operating procedures.

6. Reports

6.1 Data Packet Organization

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

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

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

6.2 Control Chart(s)

The recovery values for ICP analytes Al, B, Cd, Cr, and Ag in the LCS are plotted on control charts. Other analytes may be added at the discretion of QA without immediate revision of this SOP.

6.3 References:

Test Methods for Evaluating Solid Waste, SW-846, Method 6010B, Revision 2, December 1996

Appendix I

QC Summary Table

Laucks Testing Laboratories

Method 6010B QA Requirements and Corrective Actions					
QA Element	**Method Criterion**	**Laucks Criterion**	**Frequency**	**Corrective Action**	**Documentation**
Initial Calibration	One standard and a Blank	One standard and a Blank	Beginning of each run.		In raw data.
Initial Calibration Verification	±10% of nominal value. Made from an independant source.	±10% of nominal value. Made from an independant source.	Beginning of each run immediately following ICAL.	Recalibration required.	Form 2A or in raw data.
Continuing Calibration Verification	±10% and RSD < 5% for replicate integrations.	±10% and RSD < 5% for replicate integrations.	Every 10 samples. Mid-range	Recalibrate and rerun affected samples.	Form 2A or in raw data.
Instrument Blank	±3 sigma of average.	< 1/2 reporting limit	Every 10 samples.	Recalibrate and rerun affected samples.	Form 3 or in raw data.
Method Blank	<MDL or <5% of the concentration of any sample or <5% of the regulatory limit (whichever is highest).	< one-half the reporting limit or <5% of the regulatory limit or <5% of the sample conc (whichever is highest).	One/batch	Flag data per QCO or redigest samples	Form 3, in raw data, or database report.
Laboratory Control Sample		LCSW: 80%-120% LCSS: Manufacturer Specs.	One/batch	Redigest samples	Form 7, in raw data, or database report
Matrix Spike Recovery	75-125% or within documented historical limits	75-125% or current QC database criteria.	5% or per batch	Consult QCO	Form 5, in raw data, or database report

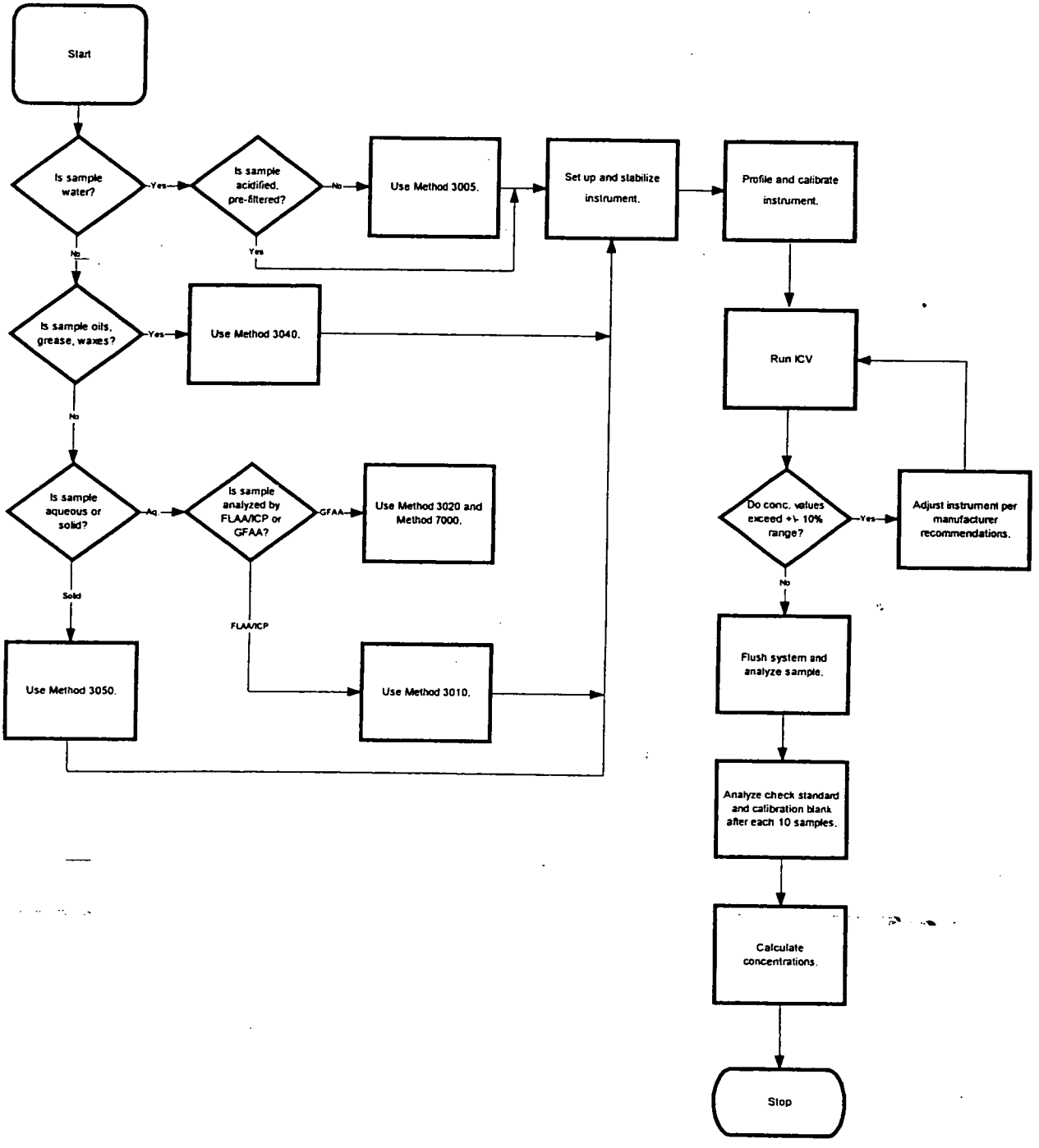
Laucks Testing Laboratories

Method 6010B QA Requirements and Corrective Actions

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Post Digestion Spike Recovery	75-125% used for new or unusual sample matrices	75-125%. Spike level either 2x RL or 2x native sample level, whichever is greater	used when MS is out of control	none	Form 5B, in raw data, or database report
Duplicate % Difference	± 20% if sample conc >10x the IDL	± 20% or current QC database criteria. ±RL if sample is <10x the IDL	5% or per batch	Consult QCO	Form 6, in raw data, or database report
Serial Dilution	± 10% difference used for new or unusual matrices	± 10% difference if sample conc > 50x the MDL	One/batch	Flag data with an "E".	Form 9 or in the raw data.
ICSA,AB	±20% true value of analytes, or ± the CRDL.	±20% true value of analytes, or ± the CRDL.	Beginning and end of run	Recalibrate and rerun affected samples	Form 4, or in raw data.
Standard Reference Material (SRM) Recovery	See QC control catalog	Control limits set by vendor	5% or per batch	Redigest samples	Form 7, in raw data, or database report

Appendix II

Flow Chart


Start
Is sample water?
Yes
Is sample acidified, pre-filtered?
No
Use Method 3005.
Yes
Set up and stabilize instrument.
Profile and calibrate instrument.
No
Is sample oils, grease, waxes?
Yes
Use Method 3040.
Run ICV
No
Is sample aqueous or solid?
Aq
Is sample analyzed by FLAA/ICP or GFAA?
GFAA
Use Method 3020 and Method 7000.
Do conc. values exceed +/- 10% range?
Yes
Adjust instrument per manufacturer recommendations.
No
Solid
FLAA/ICP
Use Method 3050.
Use Method 3010.
Flush system and analyze sample.
Analyze check standard and calibration blank after each 10 samples.
Calculate concentrations.
Stop

Appendix III

Sample Handling and Preservation

Metals (except hexavalent chromium and mercury):

Measurement	Digestion Volume Required.	Collection Volume	Preservation/Holding Time
Total recoverable	100 mL	600 mL	HNO_3 to pH <2 6 months
Dissolved	100 mL	600 mL	Filter on site; HNO_3 to pH <2 6 months
Suspended	100 mL	600 mL	Filter on site 6 months
Total	100 mL	600 mL	HNO_3 to pH <2 6 months

Solid samples should be at least 200 g and usually require no preservation other than storing at 4°C until analyzed. Either plastic or glass containers may be used for sample collection.

Appendix IV

Analytical Sequence

Standard 0
Standard 1
ICV
ICB
CRDL Check
Linear Range Check
CCV
CCB
ICSAI
ICSABI
Sample #1
Sample #1D
Sample #1S
Sample #1L
Sample #2
Sample #3
Sample #4
ICSAF
ICSABF
CCV
CCB

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-7202

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

Revision history:

Number	Date
1.0	04/26/95
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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

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1. Introduction and Scope

1.1 Method Description

Inductively coupled plasma-mass spectrometry (ICP-MS) is a technique which is applicable to µg/L concentrations of a large number of elements in water and wastes after appropriate sample preparation steps are taken. When dissolved constituents are required, samples must be filtered and acid preserved prior to analysis. No further digestion is required prior to analysis for dissolved elements. Acid digestion prior to filtration and analysis is required for groundwater, aqueous samples, industrial waste, soil, sludge, sediment, and other solid waste for which total (acid-leachable) elements are required.

See Appendix V for reporting limits.
See Appendix I for analytical masses and standard concentrations.

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

1.2 Sample Collection, Sample Storage, Holding Times

Water samples should to be collected in plastic or Teflon containers and preserved to a pH < 2. A one liter sample bottle is sufficient volume for analysis. Soil samples do not require preservation but need to be stored at 4° C and may be collected in glass if plastic containers are not available. At least 200 grams of sample should be collected. The holding time for metals is 6 months. If mercury is being analyzed by this technique, which is not currently approved or done without specific client arrangement, the holding time is 28 days.

1.3 Definition of Terms

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

Batch Identifier - A number given to each analysis group which uniquely identifies that batch. This number is preceded by an "A", mmddyy, ICPMS, matrix (W for water, S for soil), sequence number (i.e. A022595ICPMSW01).

CCB - Continuing Calibration Blank - This is the same acronym used in the CLP program. This is a blank which is analyzed immediately after the CCV (almost always after every 10 samples and at the end of the analytical run) during the analysis sequence to determine whether the instrument or system has maintained a stable baseline.

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

CLP - Contract Laboratory Program - The USEPA program that contracts with laboratories to provide laboratory services. The term has come to mean a much broader set of methods and deliverables. In context of this SOP, CLP means procedures or operations which are detailed in the CLP contract and which are extended to a broader working definition.

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

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

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

IDL - Instrument detection limit. IDL's can be estimated by analyzing seven replicates of a standard analyte solution over three nonconsecutive days. The analyte concentration should be 3-5 times the estimated IDL. Multiplying the average standard deviation by three will yield the IDL for that analyte. Each measurement must be performed as though it were a separate analytical sample. IDL's must be determined quarterly

MDL - Method detection limit - The lowest concentration of an analyte which will yield a positive result that is greater than zero at a known level of confidence. MDLs are empirically determined and are performed annually.

LCS - Laboratory Control Sample. This is a material of approximately the same matrix as the samples, containing a known and usually certified amount of target analyte and which is prepared and analyzed in the same manner as a typical sample. This

sample is used to demonstrate that the analytical system is in control. It may be considered to be a blank spike for most inorganic analyses and is preferred over artificially spiking blank materials.

Serial Dilution - If the analyte concentration is within the linear range of the instrument and is sufficiently high (minimally, a factor of 100 above the IDL/MDL), an analysis of a fivefold dilution must agree within ±10% of the original determination. If not, an interference effect must be suspected. One serial dilution must be analyzed for each twenty samples or less of each matrix in a batch. A serial dilution is denoted in the raw data by an "L".

ICSA - Interference Check Solution A. The ICSA is a solution that contains the interfering analytes. This solution is analyzed to indicate if a high level of interfering compounds will have an affect on the analytes of interest.

ICSAB - Interference Check Solution AB. The ICSAB is a solution that contains the interfering analytes and the analytes of interest. This solution is analyzed to indicate if a high level of interfering compounds will have an affect on the recovery of the analytes of interest.

Internal Standards - Internal standards are added to all blanks, standards, and samples. They monitor the affect of a sample's matrix on the quantification of the analytes of interest. The internal standards used are Sc45, In115, and Bi209.

Post-Digestion Spike - An analyte spike added to a portion of a prepared 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 concentration of the blank of any analyte of concern should not be higher than the highest of either:

(1) The reporting limit, or
(2) Five percent of the regulatory limit for that analyte, or
(3) Five percent of the measured concentration in the sample.

Corrective Action:

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

4.10 Laboratory Control Sample

The LCS is made from an independent source of the same matrix (soil or water) and is carried through the entire digestion procedure. An LCS is performed with each digestion batch. At a minimum, LCSW(water) control limits are 80% to 120%.

LCSS(soil) control limits are supplied by the manufacturer. LCSS control limits are not derived by the laboratory due to the small number of data points available from each lot of certified material.

Corrective Action

If the LCS is not within the required control limits, a redigestion will occur for the affected analytes.

4.11 Matrix Spike

A sample is chosen at random from the samples to be analyzed, and an aliquot of spiking solution is added to this sample prior to preparation. The analyst should attempt to avoid selecting samples which are identified by the client as blanks. As the purpose of the matrix spike is to test the system under "typical" conditions, the analyst may also avoid selecting the most difficult sample of the batch for spiking. The minimum frequency for MS analysis is 1 each per 20 samples per matrix. This will be best accomplished by running one with every batch for many analyses. This matrix spike sample is used to evaluate the matrix effect of the sample upon recovery of the analytes. The recovery of spike analytes is calculated as follows:

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

where:

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

For ICP/MS, control limits for spike recoveries will be 75-125% unless 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

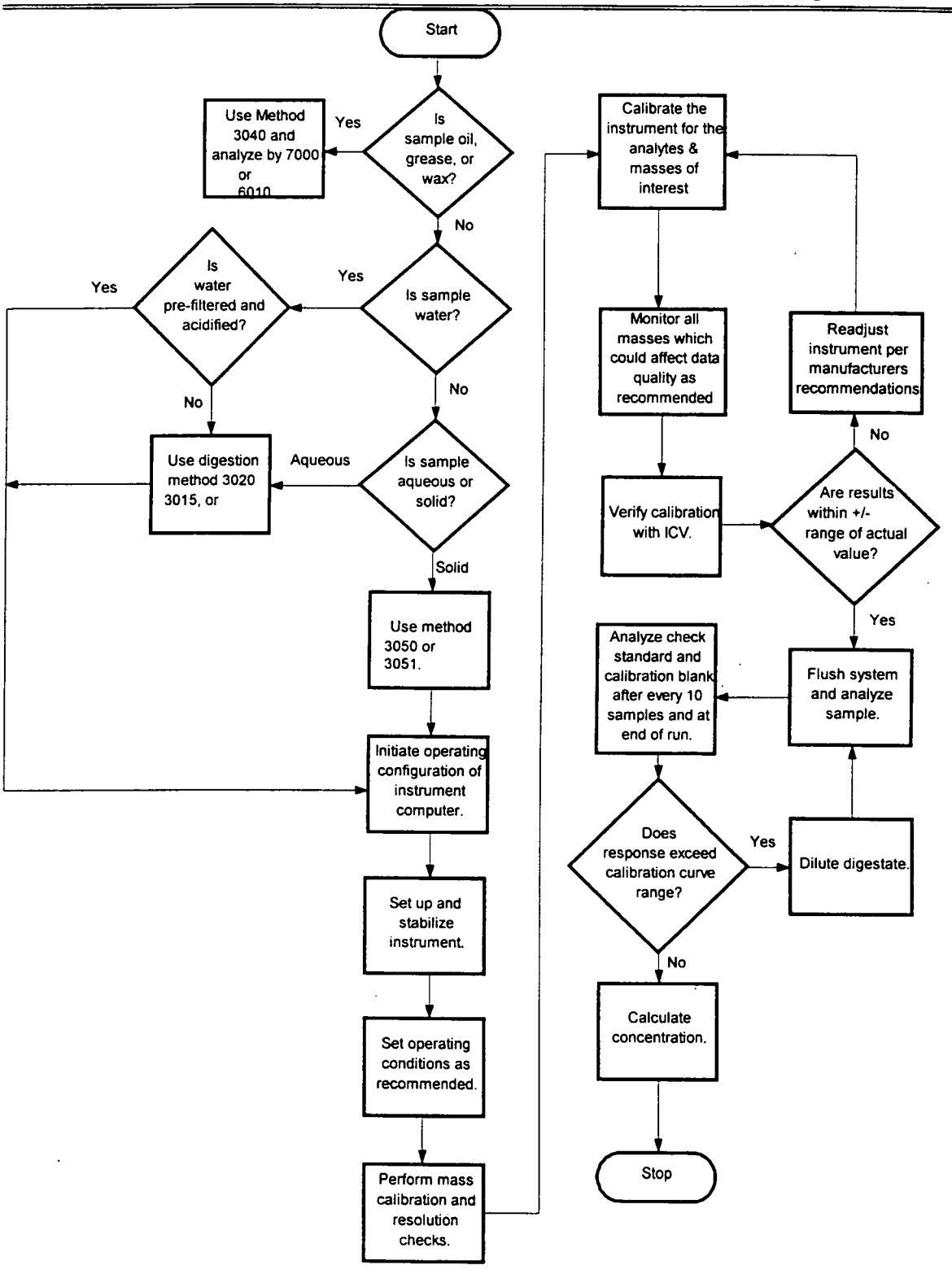
Method SW 846 6020 QA Requirements and Corrective Actions

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-7501

Title: **Operation of the PE FIMS 400 for the Analysis of Hg**

Revision history:

Number	Date
1	02/04/98
0	02/14/96

UNCONTROLLED

Revised by: Bill Lundberg Date: 2/5/98
Bill Lundberg, Inorganics Division Manager

Approved by: Harry Romberg Date: 2-5-98
Harry Romberg, QC Officer

Approved by: Karen J. Kotz Date: 2/5/98
Karen Kotz, Laboratory Director

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1. Introduction and Scope

1.1 Method Description:

1.1.1 This SOP covers the cold vapor analysis of Hg by the Perkin-Elmer Flow Injection Mercury System, Model 400 (FIMS 400). Sample handling and preparation are as required by SW 846 methods 7470 and 7471. This SOP is also valid for the preparation and analysis of samples under CLP protocol when used in conjunction with the SOW ILM04.0. Where the requirements of these procedures differ, the most stringent requirements are used. The requirement of analyzing soil samples in triplicate under 7471 is not used except on request.

1.1.2 After preparation and digestion to convert Hg to its ionic form, 0.5 mL of sample is introduced into a stream of carrier solution (3% HCl), mixed with a $SnCl_2$ suspension to reduce the Hg ion to its metallic form, which is then carried into an adsorption tube through which passes light at a wavelength of 253.7 nm. The adsorption is then measured.

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

1.2 Sample Collection, Sample Storage, Holding Times

1.2.1 Samples are stored either in glass or plastic. The holding time is 28 days from collection. Soil samples are stored at 4° C±2° C. Water samples are stored at room temperature. Water samples are preserved by the addition of HNO_3 to a pH <2.

1.3 Definition of Terms

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

1.3.2 LFB - Laboratory Fortified Blank - A sample of DIW to which a known amount of Hg is added.

1.3.3 CCB - Continuing Calibration Blank - This is the same acronym used in the CLP program. This is a blank which is analyzed immediately after the CCV (almost always

after every 10 samples and at the end of the analytical run) during the analysis sequence to determine whether the instrument or system has maintained a stable baseline.

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

1.3.5 CLP - Contract Laboratory Program - The USEPA program that contracts with laboratories to provide laboratory services. The term has come to mean a much broader set of methods and deliverables. In context of this SOP, CLP means procedures or operations which are detailed in the CLP contract and which are extended to a broader working definition.

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

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

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

1.3.9 ICV - Initial calibration verification - is a standard which is analyzed at the start of each analytical run that is compared to the initial multi-point calibration to determine whether the instrument calibration is accurate. This verification standard is from a source different from that used to make the calibration standards

1.3.10 IDL - Instrument detection limit. The lowest concentration of a target analyte that will yield a signal:noise ratio of least 3x. Determined quarterly using blanks to which a known quantity of standard has been added. The IDL is determined by analyzing 7 replicates spiked at 2-5 times the expected IDL on three non-consecutive days. The sum of the standard deviations of the three runs is the IDL.

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

1.3.12 MDL standard - Method detection limit standard - A standard prepared so that the concentration of Hg is 1-5 times the anticipated MDL

1.3.13 LCS - Laboratory Control Sample - A sample containing a known quantity of Hg used to verify digestion and analysis. LCSW indicates a water sample. It is used interchangeably with ICV. LCSS indicates a soil sample.

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

1.3.15 PBW - A blank carried through the sample preparation and digest procedures.

2. Equipment, Standards and Reagents

2.1 Equipment

2.1.1 Equivalent equipment may be used where appropriate.

FIMS 400 automatic Hg analyzer.
Perkin Elmer AS-90 autosampler.
Epson LQ300 dot-matrix printer.
Wells 18"X24" hot water bath.
BOD bottles
Analytical balance
Micropipettes: 25, 50, 100, 200, 500 and 1000 µl.
50 mL and 15 mL centrifuge tubes - polystyrene.

2.2 Standards

2.2.1 All standards must be prepared in 0.15% HNO_3.

2.2.2 Intermediate Hg solution: Dilute 100 µl of 1000 ppm Hg standard solution to 100 mL in DIW to which 0.15 mL HNO_3 has been added. The identification number of the standard solution used and the dilution must be entered on the digestion log (Appendix II). This solution must be prepared daily. Since the intermediate and working standards are prepared daily, they are not recorded in the standards logbook.

2.2.3 0.2 µg/L standard - Place 100 mL DIW in a BOD bottle. Add 20 µl stock Hg solution

2.2.4 0.5 µg/L standard - Place 100 mL DIW in a BOD bottle. Add 50 µl stock Hg solution.

2.2.5 1.0 µg/L standard - Place 100 mL DIW in a BOD bottle. Add 100µl stock Hg solution.

2.2.6 2.0 µg/L standard Place 100 mL DIW in a BOD bottle. Add 200 µl stock Hg solution.

2.2.7 5.0 µg/L standard - Place 100 mL DIW in a BOD bottle. Add 500 µl stock Hg solution.

2.2.8 10.0 µg/L standard - Place 100 mL DIW in a BOD bottle. Add 1000 µl stock Hg solution.

2.2.9 ICV solution - Add 100 mls DIW to a BOD bottle. Add sufficient stock ICV solution to produce a concentration of 1.0 to 5.0 µg/L.

2.2.10 CCV/CCB/LFB - In setting up the run, use the 5.0 µg/L standard for the CCV and LFB; for the CCB use the calibration blank.

2.2.11 MS/MSD - Add 100 µl intermediate Hg solution to the samples being spiked. When performing Hg under CLP protocols use 100 µl intermediate Hg solution and perform MS/Duplicate.

2.2.12 LCSS - Laboratory control soil sample. Concentrations and limits are certified by the manufacturer.

2.3 Reagents:

2.3.1 DIW - deionized water free of impurities.

2.3.2 H_2SO_4 (sulfuric acid) - concentrated, reagent grade

2.3.3 HNO_3 (nitric acid) - concentrated, reagent grade

2.3.4 HCl (hydrochloric acid) - concentrated, reagent grade

2.3.5 Stannous chloride - Add 11 g. $SnCl_2 \cdot 2H_2O$ (stannous chloride dihydrate) to 1 L DIW to which has been added 30 mL concentrated HCl.

2.3.6 $KMnO_4$ (potassium permanganate) - Dissolve 100 g. $KMnO_4$, marked "suitable for Hg determination", in 2 L DIW. This is a saturated solution. Sufficient undissolved $KMnO_4$ should be allowed remaining in the bottom of the bottle to assure a saturated solution.

2.3.7 $K_2S_2O_8$ (potassium persulfate) - Dissolve 50 g. $K_2S_2O_8$, marked "suitable for Hg determination", in 1 L DIW. This is a saturated solution. Sufficient undissolved $K_2S_2O_8$ should be allowed remaining in the bottom of the bottle to assure a saturated solution.

2.3.8 $NaCl/NH_2OH \cdot HCl$ (sodium chloride/hydroxylamine hydrochloride) - Dissolve 240 g NaCl (table salt is generally used) and 240 g. $NaCl/NH_2OH \cdot HCl$ (reagent grade marked "suitable for Hg determination") in 2 L DIW. Contamination has been experienced with this solution. For this reason a 50% solution should be analyzed by the FIMS 400 whenever a new bottle is made up. If the absorbance (peak height) exceeds .004 the container of $NaCl/NH_2OH \cdot HCl$ should be marked "Contaminated" and disposed of.

2.3.9 Carrier solution - The carrier is a 3% HCl solution.

3. Safety Precautions and Waste Disposal:

3.1 Safety Precautions

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

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

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

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

3.1.5 Because of the toxic nature of Hg vapor, inhalation or skin contact should be avoided.

3.1.6 The usual precautions should be taken in handling acids.

3.1.7 $SnCl_2$ is a skin and eye irritant; avoid contact.

3.1.8 The addition of hydroxylamine after digestion releases gas, including chlorine. Avoid inhalation; use hood if necessary.

4. Calibration and Quality Control

4.1 Method Detection Limit Study (MDL).

4.1.1 MDL studies for Hg water samples and soil samples are performed annually. Consult the current SOP on MDL determinations for specifics.

4.2 Initial Multi-Point Calibration:

4.2.1 Concentrations:

Standard Name	**Concentration**
S0	0.0 µg/L
S0.2	0.2 µg/L
S0.5	0.5 µg/L
S1.0	1.0 µg/L
S2.0	2.0 µg/L
S5.0	5.0 µg/L
S10.	10.0 µg/L

Standards are made fresh daily from the intermediate standard in 0.15% HNO_3.

4.2.2 The calibration blank, the ICV, the LCS, the LFB, the ICB and all standards are digested along with the samples being analyzed.

4.3 Calibration Criteria

4.3.1 Calibration curve - CC must be greater than 0.995.

4.3.2 ICV - For CLP analyses the limits are 80-120 %, for SW 846 the limits are 90-110%.

4.3.3 ICB - Limitations presently in use are -0.2 µg/L to 0.2 µg/L .

4.3.4 A PBW and an LFB are run immediately following the successful calibration. The limitations on the PBW are the same as the limitation on the ICB. The limitations on the LFB are as set forth in QC-DB.

4.3.5 If the above criteria are not met, recalibration must be performed.

4.4 Continuing quality control:

4.4.1 A CCV followed immediately by a CCB must be run every 10 samples and after the last sample. The ICB, PBW and LFB count as samples for this purpose.

4.4.2 Criteria - For CCV's the limitations are 80% to 120%. For blanks the limitations are -0.2 µg/L to 0.2 µg/L .

4.4.3 Corrective action - If a CCV or CCB are out of control, a recalibration must be performed followed by a reanalysis of all samples since the last previous successful CCV and/or CCB.

4.5 Matrix Spike

4.5.1 A sample is chosen at random from the samples to be analyzed, and 100 µl of intermediate standard is added. The spike level is then 1.0 µg/L. The analyst should attempt to avoid selecting samples which are identified by the client as blanks. As the purpose of the matrix spike is to test the system under "typical" conditions, the analyst may also avoid selecting the most difficult sample of the batch for spiking. It is not always required that a matrix spike analysis be performed with each preparation/analysis batch, however, the minimum frequency for MS analysis is 1 each per 20 samples per matrix. This will be best accomplished by running one with every batch for many analyses. This matrix spike sample is used to evaluate the matrix effect of the sample upon recovery of the analytes. The recovery of spike analytes is calculated as follows:

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

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

4.5.2 The recovery criteria are listed in QC_DB and will change from time to time.

4.5.3 Corrective action

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

4.6 Matrix Spike Duplicate

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

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

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

4.6.2 RPD control limits are listed in QC_DB and will change from time to time.

4.7 Sample Duplicate

4.7.1 Sample duplicates are required when CLP practices are employed, or when the method specifically calls for duplicates. At least one duplicate sample per 20 samples per matrix is required when matrix spikes are being performed.

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

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

4.7.2 The RPD control limits listed in QC_DB and will change from time to time.

4.7.3 Corrective action

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

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

5. Operation Procedures:

5.1 Digest - BOD bottles - Soil or Water:

5.1.1 Digestion log (Appendix II): The digestion log should show the sample numbers, the date, the name of the analyst, the time placed in the hot water bath, the time when the temperature reached 95± 5° C, and the time removed from the hot water bath. One should be prepared for each run. An example is attached as Appendix II. The numbers of each BOD bottle must be placed in the leftmost column of this form to identify which sample is in each BOD bottle.

5.1.2 Preparation of standards:

5.1.2.1 Prepare an intermediate standard daily by adding 100 µl of the 1000 mg/L stock standard to 100 mL of 0.15% HNO_3 in DIW, giving a concentration of 1.0 mg/L of Hg.

5.1.2.2 Place 100 mL DIW in each of 9 BOD bottles.. Mark these BOD bottles with the concentration of each standard in µg/L , plus one marked "Blank", one marked "ICV" and one more marked ICB.

5.1.2.3 Add from the intermediate standard the following amounts to the following BOD bottles:

0 µg/L	0 µl
0.2 µg/L	20µl
0.5 µg/L	50 µl
1.0 µg/L	100 µl
2.0 µg/L	200 µl
5.0 µg/L	500 µl
10.0 µg/L	1000 µl

5.1.2.4 To the ICV BOD bottle add that amount of the ICV standard which will produce a concentration from 1.0 µg/L to 5.0 µg/L .

5.1.2.5 The standards, the ICV and the ICB must be digested along with the PBW, LFB and samples.

5.2 Digestion - Water Samples

5.2.1 Place 100 mL DIW in the PBW BOD bottle.

5.2.2 Place 100 mL DIW + 100 µl intermediate standard in the LFB BOD bottle.

5.2.3 Place 100 mls sample in the BOD bottle designated for that sample, being sure to place 100 mL sample in the designated duplicate, MS and MSD BOD bottles for the sample involved. TCLP samples and spikes should be diluted 20 mL/100 mL (dilution factor of 5) in DIW. The TCLP spikes should be diluted 2 mL/100 mL in DIW.

5.2.4 Add the required quantity (See Sec. 4.5.1) of intermediate standard to the MS and MSD BOD bottles.

5.2.5 Add the following reagents to each BOD bottle:

5 mL concentrated H_2SO_4
2.5 mL concentrated HNO_3
15 mL $KMnO_4$ solution
8 mL $K_2S_2O_8$ solution

5.2.6 The $KMnO_4$ color must persist for 15 minutes. If it does not, more $KMnO_4$ must be added.

5.2.7 Place all the BOD bottles, being careful to keep them in order, in the hot water bath in a hood, and record the time on the digest log. Monitor the temperature of the hot water bath and record the time when the temperature reaches 90° C. Continue to heat the water bath, keeping the bath at 95 ± 5 C for two hours.

5.2.8 Remove the BOD bottles from the hot water bath and allow to cool.

5.2.9 Add 6 mL $NaCl/NH_2OH{\cdot}HCl$ solution and mix until the mixture clears. If some undissolved permanganate remains, it will usually disappear on standing after a few minutes.

5.3 Digestion - Soil samples:

5.3.1 Place 100 mL DIW in the PBW BOD bottle.

5.3.2 Place approximately 0.5 g., or, for certain CLP and 846 analyses, approximately 0.2 g of sample in each of the BOD bottles to which that sample applies. Record the exact weight on the digestion log.

5.3.3 Add 3.75 mL HCl , 1.25 mL HNO_3 and 5 ml DIW to each BOD bottle.

5.3.4 Place all BOD bottles in a hot water bath in a hood that is at 95° C ± 5° C. Maintain the heat at this level for 2 minutes.

5.3.5 Cool for a few minutes and add 50 mL DIW and 15 mL $KMnO_4$. Mix.

5.3.6 The $KMnO_4$ color must persist for 15 minutes. If it does not, more $KMnO_4$ must be added.

5.3.7 Place all BOD bottles in a hot water bath that is at 95° C ± 5° C. Record the time on the digest log. Maintain the hot water bath at this temperature for 30 minutes. Remove the BOD bottles and allow to cool.

5.3.8 Add 6 mL $HCl/NH_4NH_2OH{\cdot}HCl$ solution and mix to remove color.

5.3.9 Add 50 mL DIW. Allow to cool.

5.4 Analysis on FIMS 400.

5.4.1 This is a programmed analysis. The method name is "hg_cv" (Appendix V). This is a 6 point curve with a blank correction. The development of the calibration curve, including the ICV and ICB, is automatic. If the calibration is unsuccessful, recalibration is attempted. If a standard is seriously out of line, it is ignored by the Perkin-Elmer software. The 5.0 µg/L standard is used for CCV's; the calibration blank for CCB's, both of which are programmed to be run every 10 samples. If a CCV or a CCB fails to meet quality control specifications, the system recalibrates and reruns all samples since the last preceding valid CCV or CCB. A CCV and a CCB are also run after the last sample in the run.

5.4.2 Sample Information sheet - Appendix IV.

5.4.2.1 Click the automatic analysis button on the screen which appears after activating the program.

5.4.2.2 Click the "Sample Information" button on the toolbar.

5.4.2.3 A blank sample information sheet appears. Previous sample information sheets may be obtained by activating the File - Open menu, designating "Sample Information" from the dialog which appears. The sample information sheet should be filled in to tell the computer which auto-sampler locations to go to.

5.4.2.4 Sample ID column - Some typing can be saved by double clicking on the column title. On the resulting form the job order may be typed in and the sample numbers will then be incremented by 1 for the autosampler locations indicated. The sample ID's will then be filled in. Sample: "9802094-0001..0002. .".

5.4.2.5 Sample weight column - For soil samples the weight of the samples should be filled in. At the top, the volume units should be filled in "mL", the weight units "g".

5.4.2.6 Sample units column - Typing may be saved by double clicking on the column title and indicating the units on the dialog box which appears, and indicating the AS locations involved. In the case of water samples this will ordinarily by "µg/L ". For soil samples the button "weight:weight" should be clicked and the desired units specified in the dialog box selected. If the dialog box has been properly filled in, the machine is programmed to compute the proper units (e.g. mg/kg). It should be kept in mind that this is on an as received basis, not a dry basis.

5.4.2.7 User dilution column - Where a dilution is involved (e.g. TCLP analyses) the dilution factor should be entered. As in the sample weight and sample units columns, above, typing may be saved by double clicking the column title.

5.4.2.8 The autosampler tray being used at present is "Tray B".

Sampler Location	Size	Contents
1	50 mL tube	Calibration Blank
2	50 mL tube	0.5 µg/L standard
3	50 mL tube	1.0 µg/L standard
4	50 mL tube	2.0 µg/L standard
5	50 mL tube	5.0 µg/L standard
6	50 mL tube	10.0 µg/L standard
7	50 mL tube	ICV
8	50 mL tube	ICB
9	15 mL tube	PBW or LCSS
10	15 mL tube	LFB or sample
11-109	15 mL tubes	Samples

5.4.3 All tubes should be marked with their contents before being filled. Then the tubes are filled from the BOD bottles and placed in the autosampler locations designated either by the method or by the sample information sheet.

5.4.4 At the beginning of each day of analysis, the following steps are taken to prepare the instrument:

5.4.4.1 The flows of the carrier solution and the reductant solution (the stannous chloride solution) are measured by the methodology suggested in the Perkin-Elmer handbook "Setting Up and Performing Analyses", 2.4.2-2.4.4. The rinse container (autosampler location 0) is rinsed and filled with 3% HCl solution. This is then analyzed using the location button in the analysis window of the automated analysis window, and the curve produced displayed through the curve button in the toolbar. The curve should be flat and less than 0.0001 absorbance units.

5.4.5 Analysis - The analysis is ordinarily started by clicking the "Analyze All" button in the Automated Analysis window. If only calibration is desired, the "Calibrate" button may be used. If samples are to be analyzed by an already existing calibration curve use the "Analyze Samples" button.

5.4.5.1 Once the analysis has been started the autosampler places the sampling probe in the sample/calibration cup. The peristaltic pump begins pulling sample through the sample loop. After the line has been purged, the injection valve cycles, and injects the sample into the carrier flow. This flow is then combined

with the SnCl2 and mixed. Next it goes to a liquid/vapor separator. The liquid goes to waste and the vapor is transported into the adsorption cell of the spectrophotometer. All samples and standards are measured in duplicate.

5.4.6 Run log - A run log will be printed as the analysis progresses. The print program is designed to transmit the data to be printed to the printer one page at a time. Thus several autosampler locations will be analyzed before anything appears on the printer. If anything else is printed (e.g. the sample information sheet) while the automated analysis window is open, the computer must be rebooted, or a run log will not be printed.

5.4.7 Additional samples may be analyzed after the run designated on the sample information sheet by using the "Select Location" button on the "Analyze" page of the Automated Analysis window. The autosampler locations should be filled in the resulting dialog.. The instrument will then analyze the samples indicated. This may be used to reanalyze the samples with questionable results, or to analyze diluted samples.

5.4.8 Dilution - Where analysis results are above the high standard (10.0 µg/L) an aliquot of the sample diluted with the calibration blank may be analyzed as set forth in 5.4.7 above.

6. Reports

6.1 Data Packet Organization

6.1.1 A report is prepared using the reformat procedure on the main menu. The reformatted design is entitled "Summary". It is transferred to a floppy disk, which is then imported to a spreadsheet using Microsoft Excel 5.0. This must then be formatted into the form included in Appendix III.

6.1.2 The final data packet is to include the following:

1) The report (See Appendix III).
2) The QC_DB Report Form
2) The digestion log (Appendix II).
4) The run log, if required.

6.1.3 All results for quality control tests are entered into QC_DB. A summary report of all data entered must be included in the data packet. The routine minimum is a method blank report, an MS/MSD or MS/duplicate report and a SRM/LSCW.

6.2 Data Qualifying Flags

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

6.3 Control charts

6.3.1 The recovery values for the LCS/SRM are plotted on control charts.

Appendix I - QC Summary Table

Laucks Testing Laboratories
Method 245.1,7471,7470 QA Requirements and Corrective Actions

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Initial Calibration	CC > 0.995	CC > 0.995	once per batch	recalibrate	run log
Initial Calibration Verification	90-110 % SW846 80-120% 245.1	Per the method	one per 20	recalibrate	run log, report
Initial Calibration Blank	Below R.L.	Below R.L.	one per sequence	recalibrate	run log, report
Continuing Calibration Verification	80-120 %	80-120 %	At the beginning,every 10 samples and after the last sample	recalibrate and rerun samples since last CCV	run log
Continuing Calibration Blank	Below R.L.	Below R.L.	At the beginning,every 10 samples and after the last sample	recalibrate and rerun samples since last CCB	run log
Matrix Spike Recovery	SW-846 See QC_DB. CLP 75-125%	See QC_DB	Every 20 samples	Consult QC officer. Flag data or reanalyze.	QC_DB
MS/MSD RPD	See QC_DB	See QC_DB	Every 20 samples	Consult QC officer. Flag data or reanalyze.	QC_DB
Duplicate % Difference	20% RPD CLP	See QC_DB	Every 20 samples	Consult QC officer. Flag data or reanalyze.	QC_DB

Laboratory Fortified Blank (LFB)	85-115 %	See QC_DB	Once per 20	Recalibrate	Run log
Standard Reference Material (SRM) Recovery	Soil Manufacture's limits Water See ICV	Manufacturer's limits	Once per 20	Recalibrate	Run log

Appendix - II - Sample Digestion Log

Date		Bottles into water bath (time)	
Analyst		Water bath at 95°C (time)	
Standard ID	ME-	Bottles out of water bath (time)	
ICV No	ME-	Spike = 100 µl of Std	

No.	Sample ID	Sample size g , mL	Final volume,mL	Comments	Bottle #
	S0 ppb		100 ml		
	S0.2 ppb 20 µl of Std		100 ml		
	S0.5 ppb 50 µl of Std		100 ml		
	S1.0 ppb 100 µl of Std		100 ml		
	S2.0 ppb 200 µl of Std		100 ml		
	S5.0 ppb 500 µl of Std		100 ml		
	S10.0 ppb 1000 µl of Std		100 ml		
	ICV		100 ml		
	ICB		100 ml		
	LCSW		100 ml		
	CRA		100 ml		
	PBW		100 ml		
			100 ml		
			100 ml		
			100 ml		
			100 ml		
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			100 ml		
			100 ml		
			100 ml		
			100 ml		

$NH_2OH \cdot HCl$	NaCl	$K_2S_2O_8$	$SnCl_2 \cdot 2H_2O$	$KMnO_4$
REA1-6-3	REA1-7-9	REA1-5-7	REA1-5-3	REA1-5-4

Appendix III - Example Report Form

Hg in Water and TCLP
Digested 1/7/98
Analyzed 1/8/98

Sample	Time	Value		% Rcvry	RPD	Flag	Comment
Calib Blank	11:13:29		µg/L				
S0.2	11:16:23		µg/L				
S0.5	11:19:19		µg/L				
S1.0	11:22:19		µg/L				
S2.5	11:25:17		µg/L				
S5.0	11:28:15		µg/L				
S10.0	11:31:16		µg/L				
ICV1 ME7-23-2=4	11:34:21	4.028	µg/L	101%			
ICB	11:37:21	0.001	µg/L				
CCV1=5	11:40:16	5.015	µg/L	100%			
CCB1	11:43:10	0.007	µg/L				
LCSW1	11:46:04	4.067	µg/L	102%			
CRA	11:48:57	0.226	µg/L				
PBW	11:51:55	0.003	µg/L				
12611-11	11:54:50	2.215	µg/L				did not report
12679-02	11:57:47	0.007	µg/L				
12679-02dup	12:00:44	0.017	µg/L				
12679-02ms Spk=1	12:03:42	12.842	µg/L	1284%		N	see below
12679-03	12:06:42	0.083	µg/L				
12679-04	12:09:34	-0.016	µg/L				
CCV2=5	12:12:28	5.138	µg/L	103%			
CCB2	12:15:24	0.013	µg/L				
12679-05	12:18:16	0.026	µg/L				
12679-06	12:21:11	0.029	µg/L				
12679-07	12:24:04	0.002	µg/L				
12679-08	12:26:58	8.239	µg/L				see below
12679-09	12:29:52	23.125	µg/L				did not report
12679-10	12:32:46	0.209	µg/L				did not report
12725-01	12:35:44	0.367	µg/L				
12725-02	12:38:39	-0.226	µg/L				
12725-02dup	12:41:36	-0.419	µg/L				
CCV3=5	12:44:32	4.945	µg/L	99%			
CCB3	12:47:26	0.014	µg/L				
12725-02ms Spk=200	12:50:25	199.903	µg/L	100%			
12184-5	12:53:22	1.436	µg/L				
12184-5dup	12:56:18	1.433	µg/L				
CCV4=5	12:59:16	5.111	µg/L	102%			
CCB4	13:02:10	-0.028	µg/L				
12679-02ms (rerun)	13:08:05	0.959	µg/L	96%			
12679-02ms (10x dil)	13:10:59	-0.646	µg/L				did not report
12679-08 (rerun)	13:13:49	0.001	µg/L				
12679-09 (10x dil)	13:16:40	0.211	µg/L				did not report
CCV5=5	13:19:35	4.940	µg/L	99%			
CCB5	13:22:32	0.011	µg/L				

Appendix IV - Example Sample Information Sheet

Autosampler Loading List

Sample Information File: 020598S.SIF
Methods: Soils

Location	Elements	Solution
0	Hg	Wash Solution
1	Hg	Calib Blank
	Hg	CCB1: 0.0000 µg/L
	Hg	CCB: 0.0000 µg/L
2	Hg	S0.2: 0.2 µg/L
3	Hg	S0.5: 0.5 µg/L
4	Hg	S1.0: 1.0 µg/L
5	Hg	S2.0: 2.0 µg/L
6	Hg	S5.0: 5.0 µg/L
	Hg	CCV1: 5.0000 µg/L
	Hg	CCV: 5.0000 µg/L
7	Hg	S10.0: 10.0 µg/L
8	Hg	ICV: 4.0000 µg/L
9	Hg	ICB: 0.0000 µg/L
10	Hg	Sample: CRA
11	Hg	Sample: PBW
12	Hg	Sample: LCSS
13	Hg	Sample: 9801614-01
14	Hg	Sample: 614-01ms
15	Hg	Sample: 614-01msd
16	Hg	Sample: 549-02 (10x)
17	Hg	Sample: 549-03 (10x)
18	Hg	Sample: 549-03(20x)

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP : LTL-9104

Title: **Preparation and Analysis of Total and Amenable Cyanide according to EPA Methods 335.1, 335.3, and SW 846 method 9012A.**

Revision history:

Number	Date
2	02/09/98
1	02/07/96

UNCONTROLLED

Revised by: Bill Lundberg, Division Manager Date: 2/9/98

Reviewed by: Harry Romberg, QC Officer Date: 2-9-98

Approved by: Karen Kotz, Laboratory Director Date: 2/9/98

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1. Introduction and Scope

1.1 Method Description

Cyanide in samples is determined by a preparation with manual distillation followed by an automated spectrophotometric analysis on an auto analyzer with a UV digester and in-line distillation. The routine reporting limit is 10 µg/L in waters, and 0.50 mg/kg DB in soils and sediments. Pretreatment for the determination of cyanides amenable to chlorination in waters is also included in this SOP.

This SOP is applicable to the sample preparation (for cyanide analysis) of drinking waters, surface and saline waters, domestic and industrial wastes according to EPA methods 335.1, 335.3 and SW 846 method 9012A with the main difference being the use of the in-line still and UV digestion in EPA 335.3. Also, MIDI distillation with a 50 mL water sample instead of the normal distillation of a 500 mL sample is used in this SOP. Other differences from 9012A are listed on the QA chart at the end of this SOP. Soils and sediments are MIDI distilled as a 1 g sample.

This method is restricted to use by, or under the supervision of analysts experienced in the technique described and in SW 846 methodology. Each analyst performing this method must have demonstrated the ability to perform the described analysis.

1.2 Method Deviations and Comments

The following items represent deviations of Method 9012A, as published, which are followed as standard operating procedures in the performance of this method at Laucks:

- The MIDI distillation procedure is used, exactly as written in the CLP statement of work. This distillation uses a 50 mL aliquot of sample and a final distillation volume of 50 mls. This procedure allows the lab to use less sample, and dispose of smaller volumes of waste material. All molar ratios in this distillation are identical to the method as written.

- The calibration curve specified in 9012A is reduced by 2 to compensate for the lack of sample concentration in the distillation step.

1.3 Method Summary

Sample Preparation: The MIDI distillation procedure applies to relatively solid samples such as soils, sediments and sludges, or if requested to water samples. Cyanide determined by this method is defined as cyanide ion and complex cyanides converted to hydrocyanic acid by

reaction in a reflux system with mineral acid in the presence of magnesium ion. The cyanide as hydrocyanic acid (HCN) is released from cyanide complexes by means of a MIDI reflux-distillation operation and absorbed in a scrubber containing sodium hydroxide solution. The cyanide in the distillate is then determined in a spectrophotometric analysis.

Water samples analyzed under EPA 335.3 are not externally distilled and are directly analyzed unless otherwise requested.

Spectrophotometric Analysis: On a Technicon auto analyzer, the samples are acidified and passed through a UV digester and then distilled in-line. The cyanide released is converted to cyanogen chloride, CNCl, by reaction with chloramine-T at pH less than 8 without hydrolysis to the cyanate. After the reaction is complete, color is formed on the addition of pyridine-barbituric acid reagent. The absorbance is read at 570 nm. To obtain colors of comparable intensity, it is essential to have the same salt content in both the samples and the standards.

1.4 Sample Handling, Sample Storage, Holding Times

Samples are preserved with 8 pellets of sodium hydroxide per liter of sample (pH > 12) at the time of collection. Samples must be stored at 4°C ± 2°C and must be analyzed within the specified holding time of 14 days from date of collection. The samples can be submitted in either plastic or glass containers. The pH is checked at the time of analysis and the client should be notified if the pH is less than 12.

Samples are to be checked for chlorine interference at the time of collection and the appropriate action should be taken (see section 5.4 on interferences).

1.5 Definition of Terms

This section defines terms and acronyms as they are used in this SOP. Other terms are not defined here since it is assumed that the user of this SOP already understands their more general meaning.

Batch Identifier - A number given to each preparation or analysis group which uniquely identifies that batch. This number is generally the blank ID for preparation batches only preceded by an "A" rather than a "B" for inorganic batches. The preparation batch IDs are discussed in other documentation. The batch identifier for the second set of water samples analyzed on Feb 6, 1998 would be A020698AA1W02.

CCB - Continuing Calibration Blank - This is the same acronym used in the CLP program. This is a blank which is analyzed immediately after the CCV (almost always after every 10 samples and at the end of the analytical run) during the analysis sequence to determine whether the instrument or system has maintained a stable baseline.

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

CLP - Contract Laboratory Program - The USEPA program that contracts with laboratories to provide laboratory services. The term has come to mean a much broader set of methods and deliverables. In context of this SOP, CLP means procedures or operations which are detailed in the CLP contract and which are extended to a broader working definition.

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

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

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

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

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

SRM or LCS - Standard Reference Material or Laboratory Control Sample. This is a material of approximately the same matrix as the samples, containing a known and usually certified amount of target analyte and which is prepared and analyzed in the same manner as a typical sample. This sample is used to demonstrate that the analytical system is in control.

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

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

2. Equipment, Reagents, and Standards

2.1 Equipment

Required instrumentation includes the following:

1) Midi reflux distillation system with heating block capable of maintaining 125°C ± 5°C. We use the MIDI-VAP model MC-100 ("CYN-TEN").

2) Technicon Auto-Analyzer with in-line distillation unit and UV digester.

2.2 Reagents

1) 0.25 N NaOH: dissolve 10.0 g NaOH pellets in 1 L DIW. **CAUTION**: this reagent will get hot upon mixing!

2) $MgCl_2$ solution: dissolve 510 g $MgCl_2 \cdot 6H_2O$ and dilute to 1 L with DIW.

3) 1:1 H_2SO_4: carefully and slowly add with mixing 500 mL of conc. H_2SO_4 to 500 mL of DIW. **CAUTION**: this reagent will get **EXTREMELY** hot upon mixing! Mix in a Pyrex or equivalent container! **DO NOT ADD WATER TO ACID!**

4) Phosphate buffer: dissolve 13.6 g monobasic potassium phosphate (KH_2PO_4) and 0.28 g anhydrous dibasic sodium phosphate (Na_2HPO_4) and dilute to 1 L with DIW.

5) Chloramine -T solution: dissolve 1.0 g in 250 mL of DIW. Prepare this reagent fresh daily.

6) Color reagent: place 15 g of barbituric acid in the bottom of a container, and rinse down the sides of the container with DIW. Add 100 mL of DIW and swirl gently. Add 75 mL of pyridine and mix. Add 15 mL of conc. HCl, mix, and cool. Dilute to 1 L with DIW. This reagent should be stored at 4°C. This reagent should be made under a hood.

7) Distillation reagent: to 500 mL of DIW add 250 mL conc. H_3PO_4 and 50 mL 50% H_3PO_2 and then dilute to 1 L with DIW.

8) Sodium Hypochlorite solution: Commercial bleach such as Clorox (5.5%).

2.3 Standards

(a) Stock cyanide solution for standards and spiking, 100 mg/L CN. Place 0.2510 g of KCN and approx. 1.5 g NaOH in a 1.0 L volumetric flask and dissolve. Dilute up to the mark with DIW. Record this standard in a standards logbook. The stock standard is good for one year.

(b) Intermediate cyanide standard solution, 1.00 mg/L CN. Dilute 1.0 mL of stock standard solution (a) to 100 mL with 0.25 N NaOH. Prepare fresh solution at least weekly and record in a standards logbook.

(c) Blank solution for zero standard, blanks and diluting samples and standards: Dilute 200 µL 10 N NaOH to 100 mL with DIW. Prepare fresh solution at least weekly and as needed.

(d) Calibration standards:

Concentration (mg/L)	µLs of (b)	Final volume (mL)
0.200	1000	5.0
0.150	750	5.0
0.100	500.	5.0
0.050	250.	5.0
0.020	100	5.0
0.010	50.	5.0
0	0	5.0

All standards should be diluted up to final volume with (c) Blank solution. Standards are used up in analysis so prepare fresh standards for each analytical run.

3. Safety Precautions

Refer to the Laucks SOP on lab safety practices and the instrument manufacturer's manual for routine instrument precautions.

All standards, samples and sample solutions should be handled as if they are hazardous substances. Some forms of cyanide can be released as a gaseous form upon acidification. Treat all samples as if they definitely contain cyanide.

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

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

Sodium hydroxide and sulfuric phosphoric acid are extreme irritants if they come in contact with skin or eyes. When using these chemicals, the analyst should wear gloves and safety glasses.

The color reagent (containing pyridine) should be prepared under a hood.

4. Calibration and Quality Control

Quality Control criteria are further detailed in Appendix I.

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 determining detection limits. Briefly, it involves the analysis of 7 replicate samples spiked at a concentration 1-5 times the anticipated method detection limit. A Student's T-test is then applied to these measured values to calculate the MDL. MDLs are determined annually.

4.2 Initial Multi-Point Calibration

The calibration standards listed in section 2.3 (d) should be analyzed in order from lowest concentration to highest. This calibration MUST IMMEDIATELY PRECEDE sample analysis. Initial calibration data is evaluated using the correlation coefficient of a linear regression analysis. The correlation coefficient must be 0.995 or greater. All calibration verifications and sample extract concentrations must be computed using the regression equation. If the criteria are not met, the instrument must be recalibrated.

The highest standard used in the linear regression calculation defines the upper usable working range of the instrument. Any sample concentration value that exceeds the concentration of this high standard must be diluted with blank solution (standard c) and reanalyzed.

4.3 Initial Calibration Verification

Immediately after the calibration curve, analyze an appropriate ICV. The calculated concentration of the ICV must be within ±15% of the true value. If the ICV criteria are not met, no samples can be analyzed. Perform system maintenance and re-calibrate. An LCSW is analyzed for every 20 samples per analytical run and only the first one is also an ICV.

4.4 Initial Calibration Blank

After the analysis of the ICV standard an instrument blank (ICB) is analyzed. The ICB consists of a sample of blank solution, standard (c). The levels of cyanide in the ICB must not exceed the reporting limit. If the initial ICB contains CN levels above the reporting limit, the system is out of control. The source of contamination must be identified and corrected before proceeding with the analysis. The instrument must be recalibrated.

4.5 Continuing Calibration Verification (CCV) and Blank (CCB)

The mid-range calibration standard of 0.100 mg/L is analyzed after every 10 samples (starting after the ICB) as the CCV. Immediately following the CCV, a sample of blank solution (standard (c)) is analyzed as the CCB. In addition, the CCV/CCB must be the last samples analyzed in the run.

Cyanide concentration in the CCV must fall within ± 15% of the true value. The levels of cyanide in the CCB must not exceed the reporting limit. If CCV limits are exceeded, check calculations or perform instrument maintenance. If the CCB limit is exceeded, identify the source of the contamination. No sample results may be reported that are not bracketed by a successful ICV/ICB and/or successful CCV/CCBs, and only ten samples within each successful bracket can be reported.

4.6 Preparation Blanks

For manual distillation preparation, the preparation blank (PB) consists of a 50 mL sample of DIW and is used to verify contamination free reagents and apparatus. A PB is distilled every 20 samples per analytical run. The cyanide concentration of the PB must not exceed the reporting limit. If the reporting limit is exceeded in a manually distilled PB, the lowest reportable concentration of CN in the associated samples is twenty times the PB concentration. Otherwise, all samples associated with the PB with cyanide concentrations less than twenty times the PB concentration and above the reporting limit, must be re-distilled and reanalyzed with a new PB.

For autoanalyzed water samples, a PBW is analyzed for every 20 samples (or with each SDG) analyzed. This does not include distillates from manual distillation which are represented by their distilled PB. This consists of a sample of blank solution (standard (c). If the cyanide concentration is above the reporting limit, then the blank solution is reprepared and reanalyzed as well as any samples diluted with the solution since the last PBW was checked (including the calibration). If the new PBW is in control, then the samples are verified by the new PBW. If contamination persists, then source isolated and the instrument is recalibrated and all samples are verified by the PBW are reanalyzed.

4.7 Matrix Spike

For manual distillation preparation, a sample is chosen at random from the samples to be analyzed (or a sample that is specifically requested by the client if they send excess sample volume). 50 µL of 100 mg/L CN standard solution, standard (a), is added to the 50 mL of sample prior to distillation, for a spike value of 0.100 mg/L in the 50 mL of distillate. For 9012A a 50 mL aliquot is spiked with 20 µL for a spike concentration of 0.040 mg/L The analyst must attempt to avoid selecting samples which are identified by the client as blanks. As the purpose of the matrix spike is to test the system under "typical" conditions, the analyst may also avoid selecting the most difficult sample of the batch for spiking. The minimum frequency for MS analysis is 1 each per 20 samples per matrix. This matrix spike sample is used to evaluate the matrix effect of the sample upon recovery of the analytes.

For autoanalyzed water samples, the same criteria for sample selection and frequency applies. The MS is prepared by measuring out 10 mL of the selected sample, then adding 10 µL of the 100 mg/L CN standard (standard (a)) for a spike value of 0.100 mg/L. If the sample needs dilution, the diluted sample is spiked in the same manner for a spike value of 0.100 (multiplied by the dilution factor) mg/L.

The recovery of spike analytes is calculated as follows:

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

where:

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

If the spike recovery is not at or within the limits stated in QC-DB, an action such as re-analysis of the spike at a higher dilution, or selection of an alternate sample for spiking (if possible) is

taken. An exception to this rule is granted in situations where the blank spike is in control, or the sample concentration exceeds the spike level by a factor of four or more. In these cases, a narration is included with the data.

4.8 Matrix Spike Duplicate

For both manual distillation and autoanalysis; at least one duplicate sample per 20 samples per matrix is required. RPD values are calculated as follows:

$$RPD = \frac{|SR1 - SR2|}{(SR1 + SR2)/2} * 100$$

where:

SR1 = measured concentration in the initial spike analysis
SR2 = measured concentration in the duplicate spike analysis

The recovery and RPD must fall within the control limit stated in QC-DB.

4.9 Blank Spike

To verify the manual distillation procedure, a 50 mL sample of DIW is spiked with 50 µL of the 100 mg/L standard (standard (a)) for a spike value of 0.100 mg/L, and is distilled and analyzed with the other samples. The calculated recovery must be within the limits stated in QC-DB. The minimum frequency for the BS is one per 20 samples per analytical run.

4.10 Sample Duplicate

4.10.1.1 Sample duplicates are required. At least one duplicate sample per 20 samples per matrix is required when matrix spikes are being performed. RPD values are calculated in a manner similar to MS/MSD RPDs:

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

where:

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

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

4.10.2 Corrective action

4.10.2.1 If a trend in out of control RPD values is observed, the methods used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable reproducibility. Generally, if recoveries are in control and no analyte of interest was detected in any of the samples, no immediate action will be taken on that sample set. If integrity of reported sample values is in doubt, re-analysis may be called for. Corrective actions should be discussed with the Quality Control Officer.

5. Operation procedures

5.1 Pre-distillation Sample Screen for Waters

Water samples are screened for sulfide and chlorine interferences prior to distillation as follows:

1) Pour a small amount of pH 4 buffer into a 1oz. plastic cup.

2) Check for the presence of sulfide with lead acetate test paper. Wet the paper with pH 4 buffer and then drop ~10 µL of sample onto it. A darkening of the paper indicates the presence of S^{2-}. If present then see Section 5.3 Interferences before proceeding with the distillation.

3) Check for the presence of chlorine with potassium iodide-starch test paper. Wet the paper with pH 4 buffer and then drop ~10 µL of sample onto it. A bluish discoloration indicates the presence of chlorine. If present then see Section 5.3 Interferences before proceeding with the distillation.

5.2 Pretreatment for Cyanides Amenable to Chlorination

Two sample aliquots are required for chlorine amenable cyanide determination. One is pretreated, the other is not pretreated. Both are prepped and analyzed and the difference between the two is the cyanide amenable to chlorination.

1.) To the sample aliquot designated for pretreatment add bleach solution dropwise until an excess of chlorine is maintained. Test for residual chlorine with KI-starch paper. Paper will turn blue.

2.) Maintain excess for one hour with continued agitation using a Teflon stir bar and a stir plate.

3.) After one hour, add ascorbic acid in small increments (~0.01 g) until KI-starch paper shows no residual chlorine and analyze.

5.3 Manual Distillation

Samples are distilled using the MIDI distillation method. The distillation procedure is as follows:

1) Remove fritted tubes from absorbers, rinse, and set aside.

2) Rinse out absorbers, and invert to drain.

3) Add 50.0 mL of a liquid sample (or 1.0 g of a solid sample) to a sample tube. For solid samples, dilute with DIW to the 50 mL mark on the tube.

4) Set up sample tubes for the appropriate QC (BS, PB, MS/MSD), but do not spike the BS or the "spiked" sample yet.

5) Add 3-4 boiling stones to each tube.

6) Insert reflux impingers into the sample tubes.

7) Fill the absorber tubes with 50.0 mL of 0.25 N NaOH, and insert the fritted tubes.

8) Place sample tubes in the back row of the heating block, and the absorbers into the front row.

9) Insert the cold-finger condensers into the top of the sample tubes.

10) Hook up all vacuum hoses.

11) Turn on water flow, and adjust flow meter to approx. the same number of samples that are being distilled (about 9-10 for 10 samples).

12) Plug in the vacuum pump.

13) Stabilize the flow rate at about 3 bubbles per second and wait for about 5 minutes.

14) Spike the BS and the MS/MSD with the appropriate spiking material.

15) Add 5 mL of 1:1 H_2SO_4 to each tube, and let mix for about 3 minutes.

16) Add 2 mL $MgCl_2$ to each tube, and rinse tubes with DIW.

17) Turn power on, and turn knob to mark.

18) When distillation is complete (10 minutes to heat to reflux, 60 mins of reflux, and 15 minutes of cooling) the heating block will automatically shut itself off. Replace tubes in tube rack, removing the fritted tubes before turning the vacuum pump off.

19) Pour off the liquid from the absorbers into a clean container, label, and store at 4°C if the distillates will not be analyzed immediately.

20) Rinse all glassware with DIW, and store fritted tubes in DIW.

5.4 Interferences

1.) Sulfide interference: Some sulfides convert CN^- to SCN^-. Add cadmium carbonate to sample until lead acetate test paper no longer darkens (treat 25 mL more than is necessary for the distillation). Yellow cadmium sulfide precipitates if the sample contains sulfide. Avoid a large excess of cadmium carbonate and a long contact time in order to minimize a loss by complexation or occlusion of cyanide on the precipitated material. Filter sample through a dry filter paper into a dry container then proceed with the distillation procedure. A narration is included in the final report. Run a standard or BS treated in the same manner with the treated samples.

2.) Chlorine interference: Chlorine decomposes most cyanides. Add ascorbic acid to the 50 mL sample ~0.02 g at a time until KI-starch test paper no longer turns bluish then add an additional 0.02 g of ascorbic acid. Proceed with the distillation procedure. A narration is included in the final report.

3.) NO_3 / NO_2 interference: Nitrates and/or nitrites can form oximes with organic compounds which can decompose under test conditions to form HCN. If high levels are suspected (e.g. fertilizers) then sulfamic acid is added to the sample to remove the interference.

4.) Surfactants. For manual distillation, the presence of surfactants may cause the sample to foam during refluxing. If this occurs, add an antifoam agent such as Baker Antifoam B Silicone Emulsion.

5.) Interferences are eliminated or reduced by the distillation procedure

6. Sample Analysis Using a Technicon Auto Analyzer

6.1 Instrument start-up and operation

The auto-analyzer should be set up for cyanide analysis according to the Laucks SOP on configuring the Technicon Autoanalyzer. Turn on power and allow heating bath to reach 150 degrees C while flushing reagent tubes with DIW. After the temperature is reached, place the reagent tubes into their proper reagents. Turn on chart drive motor and allow reagents to flow through completely. When baseline has settled, set baseline at about 90%T.

6.2 Analysis procedure

1) Prepare the 1.0 ppm working standard and the blank solution

2) From these solutions make the standard curve in 1 oz plastic cups, swirl and transfer to sampler cups, and load onto sampler.

3) Immediately after the calibration standards, load an EPA approved LCSW as the ICV. Immediately after the ICV, load the blank solution for the ICB.

4) Load standards, water samples, and distillates from manual distillation (distillates are analyzed the same as water samples) as in the following example and press the reset button on the autosampler to begin the analysis:

cup#	cup contains
1	STD 1 0 ppb
2	STD 2 10 ppb
3	STD 3 20 ppb
4	STD 4 50 ppb
5	STD 5 100 ppb
6	STD 6 150 ppb
7	STD 7 200 ppb
8	ICV/LCSW1
9	ICB
10	PBW1
11	Sample 1
12	Sample 1MS
13	Sample 1MSD
14	Next Sample
15	. . .
16	CCVx
17	CCBx

After sampling is finished and the last sample peak has been recorded, turn off the chart drive motor, put reagent tubes into wash and turn off the oil bath heater. Once the oil bath has cooled below 100 degrees C, the instrument can be entirely shut down.

6.3 Compound Quantification

The data for each sample is recorded in strip-chart form. On a bench sheet is recorded the information for each sample such as Sample ID, Cup #, and Dilution. Then the value for the Peak Ht and Baseline, as read from the strip-chart (to the nearest 0.25 units), is recorded for each sample. This data is then entered into a standard spreadsheet for the calculation of all other values. (An example of the spreadsheet is included in appendix II.)

- Percent transmittance is calculated from the formula: %T = Peak Ht/(Baseline/100) where the ratio Baseline/100 accounts for baseline drift.

- Absorbance is calculated from %T by the formula: Abs = 2-log(%T)

- The spreadsheet performs a linear regression including the calibration standards 0 mg/L up to 0.200 mg/L with x being the true value of the standards, and y being their corresponding absorbances.

- Concentration of CN^- in the samples is calculated from the linear regression curve using the formula y=mx+b. Substituting the sample absorbance as the y value and solving for x (conc. of CN^- in the same units as the standards were entered) and multiplying by the dilution factor, the formula becomes:

  conc. of CN^- = [(Abs. - y intercept) / slope] * dilution

7. Reports

7.1 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.
*	: QC limits were exceeded.

Appendix I
Laucks Testing Laboratories
Method LTL 9104 QA Requirements and Corrective Actions

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Initial Calibration	7 standards plus a blank. r ≥0.995	7 stds plus a blank, one std distilled as the BS. r ≥0.995	Beginning of run	Recalibrate if r < 0.995	In raw data.
Initial Calibration Verification / LCSW	Verify calibration every 15 samples with an independently prepared check standard.±15%.	Independent source ±15%.	1 per batch or 5% including one immediately after calibration.	Recalibrate	Enter in QC database under SRM.
Initial Calibration Blank	No criteria.	≤0.010 mg/L	1 per run immediately after ICV.	Recalibrate	In raw data.
Continuing Calibration Verification	Verify calibration with the mid-range standard. Must agree within 15% of true value.	Verify calibration with the mid-range standard. Must agree within 15% of true value.	1 after every 10 samples, and at the end of the run.	Perform instrument maintenance and recalibrate.	In raw data.
Continuing Calibration Blank	No criteria.	≤0.010 mg/L or reporting limit	1 after every 10 samples, and at the end of the run.	Check for source of contamination. Reanalyze associated samples.	In raw data.
Preparation Blank Soil: Water:	No criteria. No criteria.	≤0.50 mg/kg DB ≤0.010 mg/L or reporting limit	1 per batch or 5%.	Check for source of contamination. Reanalyze / reprep w/ associated samples.	Enter in QC database under BLANKS.
Matrix Spike (%Rec) Soil: Water:	Run one spike duplicate every 10 samples. No recovery criteria .	Refer to current control limits catalog	1 per batch or 5%	Spike at higher level or choose another sample and reanalyze / reprep w/ associated samples.	Enter in QC database under MSPKDATA.
Matrix Spike Dup. (RPD) Soil: Water:	See MS.	Refer to current control limits catalog	1 per batch or 5%	Same as MS.	Same as MS.
Blank Spike (%Rec) (Manually distilled soils and waters only.)	Two stds distilled and compared to equal values in standard curve.	Refer to current control limits catalog	1 per batch or 5%	Reprep w/ associated samples.	Enter in QC database under BS_CALC.

Appendix II

Excel spreadsheet used for cyanide calculations.
(example)

Cyanide Analysis	ITEM	DATE	METHOD	ANALYST	CHECKER	
JOB#:	prep.		Man. Distill	PL		
MATRIX: water	analysis	1/25/96	EPA 335.3	PL		

STANDARD CURVE LCSW: WP586 (tv=0.500 ppm)

standard stock:
TE4-67-17

T.V. (mg/L CN)	Peak Ht.	Baseline	%T	Absorbance
0.000	94.00	94.50	99.47	0.002
0.010	88.50	94.50	93.65	0.028
0.020	80.50	94.50	83.44	0.055
0.050	67.50	94.50	71.43	0.146
0.100	47.50	94.50	50.26	0.299
0.150	30.50	94.50	36.24	0.512
0.200	17.75	94.50	18.78	0.726
--x value--				--y value--

r = 0.9999 m = 2.9043 y(0) = 0.0022 x(0) = -0.0008

ms/msd=0.100ppm

Sample I.D.	Dilution	Peak Ht.	Baseline	%T	Absorbance	CN (mg/L)	
ICV / LCSW1	5	54.25	95.00	57.11	0.243	0.4151	83% Rec.
ICB	1	94.25	95.00	99.21	0.003	0.0004	ND
MDL tv=0.005	1	91.25	95.00	96.05	0.017	0.0053	105% Rec.
PBW1	1	94.50	95.00	99.47	0.002	0.0000	ND
5555-1	1	94.50	95.00	99.47	0.002	0.0000	ND
5555-1ms	1	48.00	95.00	50.53	0.296	0.1013	101% Rec.
5555-1msd	1	48.25	95.00	50.79	0.294	0.1005	101% Rec.
5555-2	1	94.25	95.00	99.21	0.003	0.0004	ND
5555-3	1	92.00	95.00	96.84	0.014	0.0040	ND
5555-4	1	89.00	95.00	93.68	0.028	0.0090	0.009
5555-5	1	88.50	95.00	93.16	0.031	0.0098	0.010
5555-6	1	65.75	95.00	69.21	0.160	0.0543	0.054
CCV1 tv=0.100	1	45.50	95.00	47.89	0.320	0.1093	109% Rec.
CCB1	1	94.00	95.00	98.95	0.005	0.0008	ND

Appendix III

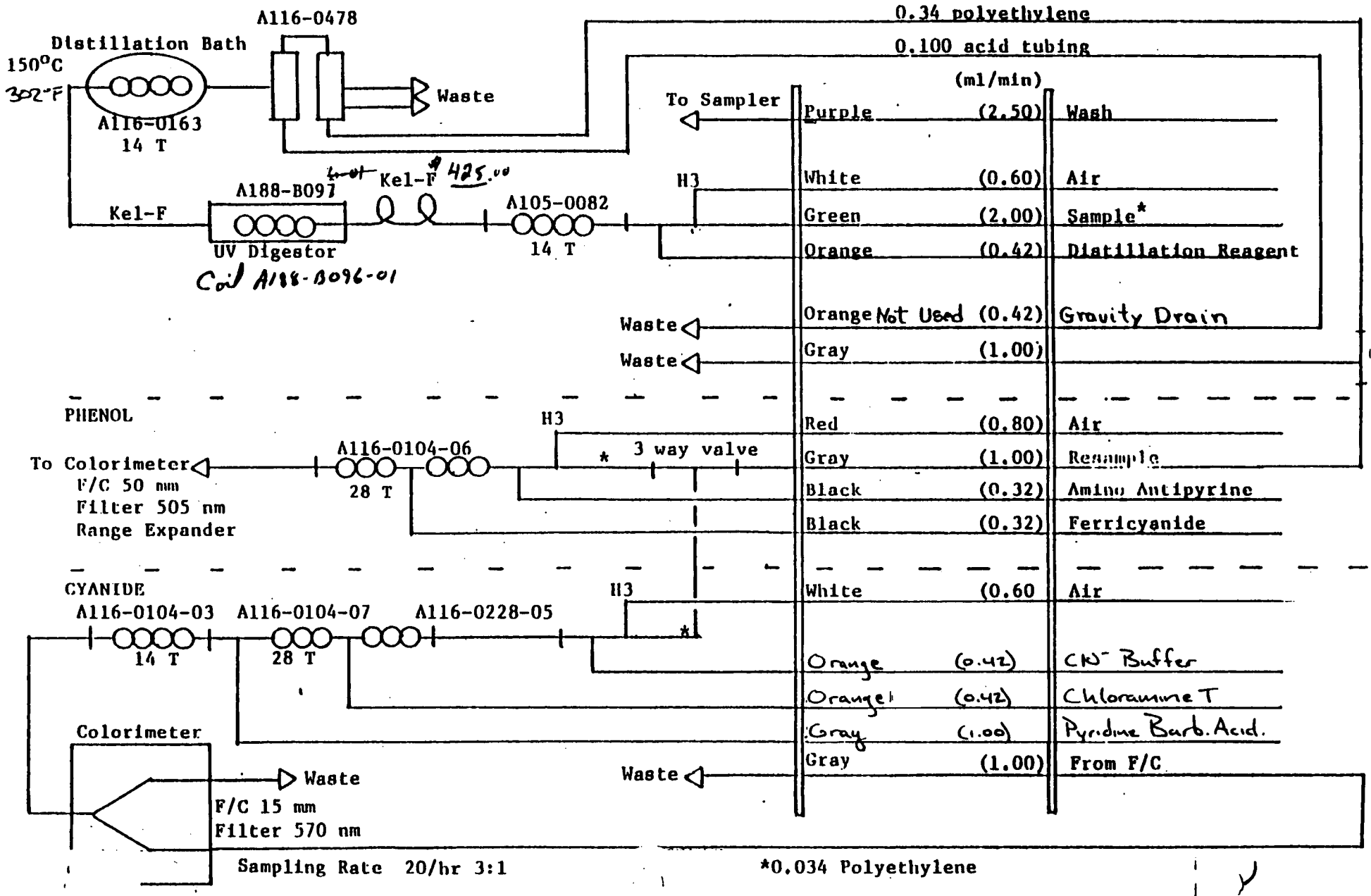
Autoanalyzer Flow Diagram

[illegible] schematic currently in use. Jan 29, 1991 MS

CYANIDE-PHENOL
Combined Manifold
AAI

ALPKEM Corporation
1981
RRP

DISTILLATION



LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-9110

Determination of Anions by Ion Chromatography by Method 300.0A or 9056

Revision history:

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UNCONTROLLED

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Approved by: Kathy Kreps, Laboratory Director — Date: 4-22-98

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1. Introduction and Scope

1.1 Method Description

1.1.1 This method describes the technique used to determine anions in water using Ion Chromatography. The method is capable of being extended to the analysis of anions in soil matrices if a preliminary leaching step is performed to solubilize the anions. Anion determinations may also be performed on the solution resulting from the oxygen bomb decomposition of fuels, solids, oils, and total/organic halogen extracts. The ions that may be quantitated by this method are fluoride, chloride, nitrite, nitrate, sulfate, bromide and phosphate.

1.1.2 The anions in an aqueous solution are introduced via an autosampler into a flowing eluant stream. This stream is passed through a cation exchange column, which strips the metallic elements from the solution. It then passes through an anion exchange column where the chromatographic separation occurs. The separation column effluent is then passed through an anion micromembrane suppresser where counter ions are exchanged for hydronium ions. This serves to suppress the conductance of the eluant and enhances the conductance of the anionic analytes of interest by forming their complementary acids. Following this exchange the stream is passed through a conductivity detector. The detector output is collected by a data acquisition system, which stores the chromatograms for later calculation and interpretation.

1.1.3 The method is based on EPA 300.0 A, or SW-846 9056. Method choice is based on client request or environmental program. The following table summarizes the differences between the two methods.

Method 300.0 Criteria	Method 9056
CCV must be ±10%	CCV must be ±5%
Duplicate frequency is 1/20	Duplicate frequency is 1/10

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

1.2 Sample Collection, Sample Storage, Holding Times

1.2.1 Samples are collected in 500 or 1000 mL unpreserved polyethylene bottles. Upon receipt, samples are stored 4° ± 2° C.

1.2.2 Analysis holding time is dependent on the anion of interest. Holding time for Nitrate, Nitrite, and Ortho phosphate analysis is 48 hrs. Holding time for Bromide, Chloride, Fluoride, and Sulfate is 28 days.

1.3 Definition of Terms

CCB - Continuing Calibration Blank - This is the same acronym used in the CLP program. This is a blank which is analyzed immediately after the CCV (almost always after every 10 samples and at the end of the analytical run) during the analysis sequence to determine whether the instrument or system has maintained a stable baseline.

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

CLP - Contract Laboratory Program - The USEPA program that contracts with laboratories to provide laboratory services. The term has come to mean a much broader set of methods and deliverables. In context of this SOP, CLP means procedures or operations which are detailed in the CLP contract and which are extended to a broader working definition.

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

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

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

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

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. See section 4.1 for a more detailed discussion of MDL determination.

SRM or LCS - Standard Reference Material or Laboratory Control Sample. This is a material of approximately the same matrix as the samples, containing a known and usually certified amount of target analyte and which is prepared and analyzed in the same manner as a typical sample. This sample is used to demonstrate that the analytical system is in control.

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

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

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

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

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

2. Equipment List and Standards

2.1 Equipment

2.1.1 Ion chromatograph, Dionex model 2000i or equivalent consisting of a dual piston pump, autosampler, either manual or automated injector, guard column, separator column, anion micromembrane suppresser, and a conductivity detector.

2.1.2 Guard column, Dionex AG4A or equivalent

2.1.3 Chromatographic column, Dionex AS4A or equivalent

2.1.4 Anion Micro Membrane Suppresser, Dionex AMMS-1 or equivalent

2.1.5 Electronic data collection device, Ezchrom data acquisition system or equivalent, or other applicable data collection system.

2.2 Standards and Solutions

2.2.1 Calibration Standard/Spiking Solution.

2.2.1.1 A 100 mL calibration standard/spiking solution is prepared as stated in the following table from vendor supplied primary standards. The primary standards are stored in the standards refrigerator at 4 °C. They are replaced annually. Upon receipt the bottles are logged into the standard logbook and are QC'd.

Analyte	Stock Conc. (mg/L)	Volume of Stock (µls)	Final Conc. (mg/L)
F	1000	500	5
Cl	1000	1000	10
NO_2 as N	100	2000	2
Br	1000	500	5
NO_3 as N	1000	500	5
PO4 as P	50	10000	5
SO_4	1000	2000	20

The above solution expires after 3 months. It is stored at room temperature. The routine calibration solution does not contain Br, and PO4. These analytes are added when required.

2.2.1.2 Spiking - Samples are spiked by adding 100µl of solution prepared in 2.2.1.1 to an aliquot of sample and diluting to 500 µl. The volume of sample, the volume of spike, and the spike reference number are recorded in the pretreatment field of the batch summary.

2.2.2 ICV Solutions.

2.2.2.1 An ICV mix for fluoride, chloride, nitrate, phosphate and sulfate is vendor purchased.

2.2.2.2 A 1. mg/L nitrite ICV solution is prepared by diluting 100 µL of a 1000 mg/L vendor supplied solution to 100 mls in a volumetric flask. This solution is prepared weekly.

2.2.2.3 A 1000 mg/L primary bromide ICV solution is prepared by diluting 0.1288 g of desiccated NaBr to a final volume of 100 mL. This solution is prepared annually Desiccated NaBr is prepared by first drying the salt in a 105°C ± 5°C drying oven for one hour. The salt is then cooled to room temperature in a desiccator.

2.2.2.4 A 5. mg/L working bromide ICV is prepared by diluting 250 µL of 2.2.2.3 to a final volume of 50 mL. This solution is prepared fresh daily.

2.2.3 Eluant Solution.

2.2.3.1 The eluant is 1.7 mM sodium carbonate and 1.8 mM sodium bicarbonate. The eluant is prepared by dissolving 0.290 g sodium bicarbonate and 0.380 g sodium carbonate in DIW and diluting to a final volume of 2 L.

3. Safety precautions and Waste Disposal

3.1 Safety Precautions

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

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

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

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

3.2 Waste Disposal

3.2.1 There is no significant waste generated from this procedure. Any remaining liquids are disposed of by flushing down the sink. Solid material may be disposed of in the garbage.

4. Calibration and Quality Control

4.1 Method Detection Limit Study

4.1.1 Prior to the analysis of any samples, it is necessary to establish method detection limits. This procedure is fully described in the Laucks SOP on the determination of method detection limits (LTL-1011). This SOP can be found in the SOP manual. Briefly, it involves the analysis of 7 replicate samples spiked at a concentration near the anticipated method detection limit. A Student's T-test is then applied to these measured values to calculate the MDL.

4.1.2 MDL studies for the ion chromatograph must be determined at a minimum of every six months, with each new analyst, or whenever there is a significant change in system response.

4.2 Determination of Retention Time Windows

4.2.1 Every six months, or any time there is a significant change in the system, the retention time windows need to be rerun

4.2.2 The RTW is determined by taking the retention times of three CCVs (mid range standards) on any one day, and computing the standard deviation. The standard deviation multiplied by 3 is the retention time window. This procedure is similar to that described in the Laucks SOP on Determination of Retention Time Windows (LTL-8000) except one run is used rather than a 72-hour period. This value is then entered into the EZ Chrom method. The retention time windows are stored in the method.

4.2.3 This data is generated on an Excel spreadsheet and is archived on the laboratory data base.

4.3 Initial Multi-Point Calibration

4.3.1 A calibration curve consists of four points and a blank. All calibration standards are diluted in sample vials at the instrument just prior to analysis as stated in the following table:

Standard Number	Volume Standard	Volume DIW	Total Volume
1	500 µl	0 µl	500 µl
2	250 µl	250 µl	500 µl
3	100 µl	400 µl	500 µl
4	25 µl	475 µl	500 µl

4.3.2 The elution order for this system is fluoride, chloride, nitrite, Bromide, Nitrate, phosphate, sulfate.

4.3.3 Samples that exceed the high standard must be diluted.

4.3.4 A multi-point calibration of the system is not required with every analytical run.

4.3.5 If the previous calibration is verified by an ICV, the calibration is considered valid and the instrument does not require re-calibration.

4.3.6 A curve is not valid for a period of greater than six months.

4.3.7 Criteria

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

4.3.8 Corrective action

4.3.8.1 If the criteria are not met, the instrument must be recalibrated. No data can be reported for an analyte that does not have a valid calibration.

4.4 Initial Calibration Verification

4.4.1 At the start of each analytical run, analyze a standard from a source other than that from which the calibration material was obtained.

4.4.2 Criteria

4.4.2.1 ***The calculated concentration of the ICV for fluoride, chloride, nitrate, phosphate and sulfate must be within the limits supplied by the manufacturer*** or not to exceed ±10% of the true value. The calculated concentration of the ICV for bromide and nitrite should not exceed 90%-110% of the true value.

4.4.3 Corrective action

4.4.3.1 If the ICV criteria are not met, no samples can be analyzed. Perform system maintenance and re-check the ICV. If the criteria still cannot be met, the system must be recalibrated. No data can be reported for an analyte that does not have a valid ICV.

4.5 Initial Calibration Blank

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

4.5.2 Corrective action

4.5.2.1 If the ICB contains target analyte levels above the reporting limit, the system is out of control. The source of contamination must be identified and corrected before proceeding with the analysis. No data can be reported for an analyte that does not have a valid ICB.

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

4.6.1 A mid-range calibration standard is analyzed at a minimum of once every 10 sample injections. Immediately following the CCV, a blank solution is analyzed. In addition, this standard and blank must be the last samples analyzed in the run.

4.6.2 Criteria

4.6.2.1 The CCV must fall within ± 10% of the true value for Method 300.0, and within ± 5% for method 9056.

4.6.2.2 The levels of target analytes in the CCB should not exceed the reporting limit.

4.6.3 Corrective action

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

4.6.3.2 If the CCB contains target analyte levels above the reporting limit, the system is out of control. The source of contamination must be identified and corrected and the affected

samples re-analyzed. As with the CCVs, no sample results may be reported that are not bracketed by a successful initial and continuing calibration blank which are in control or by preceding and following CCBs which are within limits.

4.7 Method Blanks

4.7.1 Method blanks are used to verify contamination free reagents and apparatus. They are prepared with every set of samples prepared at the same time, or at least one blank every 20 samples, whichever is more frequent. Any analyte response above the reporting limit is reported. For the purpose of water analyses, the ICB, CCB's, and the method blank are the same. Method blank control limits are that contamination should not exceed the reporting limit for waters. Method blanks should not exceed twice the reporting limit for soil leaches, oxygen bomb extracts, and total/organic halogen extracts.

4.7.2 Corrective action

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

4.8 Matrix Spike

4.8.1 Samples are spiked by adding 100µl of calibration/spiking solution to an aliquot of sample and diluting to 500 µl. The volume of sample, the volume of spike, and the spike reference number are recorded in the pretreatment field of the batch summary.

4.8.2 A sample is chosen at random from the samples to be analyzed, and an aliquot of spiking solution is added to this sample prior to preparation. The analyst should attempt to avoid selecting samples which are identified by the client as blanks. As the purpose of the matrix spike is to test the system under "typical" conditions, the analyst may also avoid

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

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

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

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

4.8.4 Corrective action

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

4.9 Sample Duplicate

4.9.1 Criteria

4.9.1.1 Sample duplicates are required when running method 300.0, 9056 or when CLP practices are employed. At least one duplicate sample per 10 samples per matrix is required when method 9056 is being performed. At least one duplicate sample per 20 is required on all other methods. RPD values are calculated in the following manner:

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

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

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

4.9.2 Corrective action

4.9.2.1 If a trend in out of control RPD values is observed, the methods used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable reproducibility. Generally, if recoveries are in control and no analyte of interest was detected in any of the samples, no immediate action will be taken on that sample set. If integrity of reported sample values is in doubt, re-analysis may be called for. Corrective actions should be discussed with the Quality Control Officer.

5. Operation procedures

5.1 Sample Analysis

5.1.1 Analysis tools and observations

5.1.1.1 If a discrete bottle number is referenced on the sample container, this information must be recorded in the pretreatment field of the batch summary.

5.1.1.2 Samples that appear overly turbid, or that have suspended or floating particles need to be filtered through a 0.45 µm syringe filter prior to analysis. If this procedure is followed, it should be documented in the pretreatment field of the batch summary

5.1.1.3 If conductivity or TDS data is available, this can be used as a tool in deciding what dilutions are appropriate.

5.1.1.4 Poor resolution between chloride and nitrite, phosphate and sulfate, and nitrate and bromide in standards or CCV's is an indication that column needs to be cleaned. If after cleaning the CCV does not resolve back to baseline between the two peaks of interest, the column has lost enough theoretical plates to render it unsuitable for analysis of those ions.

5.1.1.5 An abnormally shaped sulfate peak may indicate a sample with a high phosphate concentration. The chromatogram should be compared to one run at a greater dilution to see if peak resolution can be obtained.

5.1.1.6 The expected baseline conductivity for the system is 17.5 to 19.0 µs.

5.1.1.7 The expected back pressure of the system is 750 to 1200 psi. When the upper limit is approached the analyst should replace the eluant filter, replace the bed supports, clean the column, or reduce the flow rate. It is acceptable to reduce the flow down to 1.2 ml/min.

5.1.1.8 Other anomalies should be discussed with the departmental supervisor in order to determine appropriate corrective actions.

5.2 Analysis sequence

Calibration standards (from high to low concentration)
ICV Solutions necessary for that analytical run
ICB
10 or fewer injections
midrange standard (CCV)
DIW (CCB)
additional samples (not more than 10 injections)
additional CCV's and CCB's
...
...
must close with CCV and CCB

5.3 Instrumental Conditions

5.3.1 System Conditions

- Eluant flow is between 1.0 and 2.0 mL/min.
- Injection volume is 50 µL.
- Temperature correction factor is 1.7.
- Detector range is 30 µS.
- Analysis time is between 10 and 13 minutes.

5.4 Analytical Operation

5.4.1 Analytical procedure for liquid matrixes

5.4.1.1 A set of samples is chosen to be analyzed. Consideration should be given to holding times, due dates, and analytes of interest when choosing the sample set for a run.

5.4.1.2 A run log is constructed for the sample set. This includes the appropriate dilutions for the given samples.

5.4.1.3 The run log is programmed into the integrator or equivalent device (along with the sample dilutions), the run and sample counters are reset, and the end run statement is input.

5.4.1.4 An appropriate number of 500 µL sample vials are rinsed and placed into the auto sampler trays.

5.4.1.5 500 µL of standards, samples and QC events are placed into the sample vials.

5.4.1.6 Dilutions up to 50x can be done directly in the sample vials, greater dilutions will require an external dilution in a Solo cup or a combination of external and sample cup dilutions.

5.4.1.7 Samples that appear turbid or that have suspended particles should be filtered through a 0.45 µm syringe filter prior placing in sample vials and/or dilution. If a sample is pre-filtered, it must be documented in the pretreatment field of the batch summary.

5.4.1.8 Sample vials are capped with filter caps and placed in the auto sampler.

5.4.1.9 The "Inject A" button on the integrator and the remote button on the automation interface are pushed simultaneously to begin analysis.

5.4.2 Analytical procedure for soil or sludge matrixes.

5.4.2.1 The soil/sludge is leached with DIW by transferring 10. g to a tared 250 mL digest bottle, adding 100 mL DIW and placing on the shaker for 15 minutes or more at a shaking rate sufficient to suspend the majority of the solids. The solids are then allowed to settle and an aliquot of the supernatant is filtered through a 0.45 µm syringe filter prior to use. The leaching and filtration of the sample must be recorded in the pretreatment field of the batch summary.

5.4.2.2 A 100 mL aliquot of DIW is also shaken in a 250 mL digest bottle to be used as a method blank.

5.4.2.3 The resulting supernatant is treated as a water sample.

5.4.2.4 A total solids determination is made on all soil/sludge samples and the results are reported on a dry weight basis.

5.5 Compound Quantification

5.5.1 The data aquisition system generates a report with the chromatogram of the sample, the date analyzed, the sample identification, the method used to quantify the sample, the sample dilution, and the calculated concentration of the sample corrected for dilution.

5.5.2 For soil/sludge samples, the mg/L integrator value is converted to mg/Kg DB by the following equation

Soil:

$$Cs = \frac{Ci * Vs * 100}{Ws \; Ts}$$

Cs = Sample concentration (mg/kg DB)
Ci = Concentration in sample injected (mg/L)
Vs = Volume of extract (mL)
Ws = Weight of sample extracted (gm)
TS = Total solids (%)

6. Reports

6.1 Sample Results and Quality Control Reports

6.1.1 Data packages shall consist of the sample results summary, the chromatograms for samples in the workorder, and a copy of the QC summary report form. These include the method blank analysis, matrix spike results, duplicate results, and the Standard Reference Material (SRM) reports. A copy of the QC summary report form must be included in order to indicate that the QC was evaluated for analytical consistency.

6.2 Data Qualifying Flags

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

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

Appendix I

Laucks Testing Laboratories
Method 300.0A,9056 QA Requirements and Corrective Actions

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Initial Calibration	A minimum three point curve, that brackets the sample concentrations and a blank.	A four point curve that brackets the sample concentrations .	As instrument performance dictates, or when any change is made in the analytical system.	Fix the problem and recalibrate.	In the raw data package for all validatable packages and referenced in all other data.
Initial Calibration Verification / SRM	Not to exceed ±10% of the true value.	Nitrite and bromide to ±10% of the true value. ***Fluoride, chloride, nitrate, phosphate and sulfate values are vendor supplied*** but must not exceed ±10% of the true value.	Once per analytical run, or 1/20.	Reanalyze, if reanalysis fails recalibrate and or perform instrument maintenance. No data can be reported if ICV fails.	In the raw data package, and in the quality control data base.
Initial Calibration Blank	Value not to exceed the MDL.	Value not to exceed the Reporting Limit.	Immediately following the ICV.	Reanalyze. No data can be reported if ICB fails.	In the raw data package, and in the quality control data base.
Continuing Calibration Verification	Not to exceed ±10% of the true value.	Method 300.0: Not to exceed ±10% of the true value. ---------- Method 9056 not to exceed ± 5% of the true value.	At a minimum, after every 10 analytical samples and as the penultimate injection.	Samples which are not bracketed by in control CCV's must be reanalyzed.	In the raw data package.
Continuing Calibration Blank	Value not to exceed the MDL.	Value not to exceed the Reporting Limit.	Immediately following a CCV's.	Samples which are not bracketed by in control CCB's must be reanalyzed.	In the raw data package.

Laucks Testing Laboratories
Method 300.0A,9056 QA Requirements and Corrective Actions

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Method Blank	Value not to exceed the MDL.	ICB and method blank are the same QA event for waters. Not to exceed 2x the reporting limit for non-water matrixes.	Run at a minimum of 1 per 20 samples and as dictated by non-water matrixes.	If sample concentration exceed 10x the contamination level, no action required. Samples with contaminated Method Blanks must be re-extracted.	In the raw data package, and in the quality control data base.
Matrix Spike Recovery	Not to exceed ±20% of the true value or x ± 3S. Once every 10 samples.	To meet or exceed criteria in the current lab control limits catalog or in quality control data base.	Once every 20 samples, or per client request, whichever is more frequent.	Review data, consult QC officer, some conditions may require reanalysis.	Narrative comment in report in quality control data base, and in raw data package.
Duplicate/ Relative % Difference (RPD)	NA	To meet or exceed criteria in the current lab control limits catalog or in quality control data base.	Method 300.0: Once every 20 samples, or per client request, whichever is more frequent. ---------- Method 9056: Once every 10 samples.	Review data, consult QC officer, some conditions may require reanalysis.	Narrative comment in report in quality control data base, and in raw data package.

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #: LTL-9113

Title: **pH in Soil and Waste Samples Using SW846 Method 9045C**

Revision history:

Number	Date
1.0	07/13/94
2.0	01/16/97

UNCONTROLLED

Written by: Bill Lundberg Date: 1/16/97
Bill Lundberg, Inorganic Supervisor

Reviewed by: Harry Romberg Date: 1-16-97
Harry Romberg, QA Officer

Approved by: Karen J Kotz Date: 1/16/97
Karen Kotz, Lab Director

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1. Introduction and Scope

1.1 Method Description

1.1.1 This method is an electrometric procedure for the determination of pH in soils and waste samples.

1.1.2 The sample is mixed with deionized water (DIW). The pH of the solution is then measured with a pH meter.

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

1.2 Interferences:

1.2.1 Samples with very low or very high pH may give incorrect readings on the meter. For samples with a true pH of >10, which is not uncommon for some ash and other waste materials, the measured pH may be incorrectly low. This error can be minimized by using a low-sodium-error electrode. Strong acid solutions, with a true value of <1, an uncommon condition, may give incorrectly high measurements.

1.2.2 Temperature fluctuations will cause measurement errors. Most pH electrodes are calibrated to 25°C. Whereas Laucks does use a temperature correction probe with the pH meter, significant differences in pH can affect the solubility and apparent pH characteristics of the solution itself. Therefore, measurements should be made near the appropriate temperature by allowing the sample and diluent (DIW) to warm to room temperature prior to analysis.

1.2.3 Errors can occur when the pH electrode becomes coated. If this occurs, the electrode must be thoroughly clean and re-buffered prior to measuring the pH of subsequent samples.

1.3 Sample Collection, Sample Storage, Holding Times

1.3.1 There is no official holding time for the analysis of soil pH. The holding time for aqueous samples is to analyze immediately. Soil should not demonstrate significant pH fluctuations if refrigerated and analyzed within a reasonable time but Laucks has chosen to analyze soils samples as soon as is reasonable after collection, not to exceed 7 days from collection.

1.4 Definition of Terms

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

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

1.4.1.2 **Sequence** - A set of sample extracts and standard solutions presented to an instrument in a chronologically continuous group.

2. Equipment List, Reagents and Standards

2.1 Apparatus

pH meter with means for temperature correction (temperature correction probe)
Glass electrode
50 ml. beaker
Analytical balance capable of weighing to 0.1 g.

2.2 Standards

pH 4 buffer
pH 7 buffer
pH 10 buffer

2.3 Reagents

Deionized water (DIW)

3. Safety precautions and Waste Disposal

3.1 Safety Precautions

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

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

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

3.2 Waste Disposal

3.2.1 There should be no significant waste associated with this procedure which cannot be disposed of in the laboratory sink and/or garbage.

4. Calibration and Quality Control

4.1 Calibration

4.1.1 The pH meter must be calibrated with at least two pH buffers. One is the pH 7 buffer and the other depends upon the samples. If high pH is expected, the second buffer should be at pH 10 or higher. If low pH is expected, the second buffer should be the pH 4 buffer. High pH samples should not be analyzed from a low pH calibration and visa versa.

4.2 Sample Duplicate

4.2.1 Criteria

4.2.1.1 At least one duplicate sample per 10 samples per matrix is required. RPD values are not currently calculated for this analysis. The control limit is considered to be 0.5 pH units.

4.2.2 Corrective action

4.2.2.1 If an out-of-control duplicate pair is observed, the methods used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable reproducibility. If integrity of reported sample values is in doubt, re-analysis may be called for. Corrective actions should be discussed with the Quality Control Officer.

5. Operation procedures

5.1 Sample preparation and pH measurement of soils

5.1.1 To 20 grams of soil in a 50 ml beaker, add 20 mls of DIW and stir the suspension for 5 minutes. This may also be done with a magnetic stirring bar.

5.1.2 Allow the suspension to stand for about 1 additional hour with no agitation in order to allow most of the suspended particulates to settle out.

5.1.3 Immerse the electrode (including the temperature correction probe) just far enough into the solution to establish good electrical contact. It should not come into contact with the soil itself.

5.1.4 The electrodes should be thoroughly rinsed between samples.

5.1.5 Report the results as "soil pH measured in water @ ____ °C."

5.2 Sample preparation and pH measurement of waste material:

5.2.1 To 20 grams of waste material in a 50 ml beaker, add 20 mls of DIW and stir the suspension for 5 minutes. This may also be done with a magnetic stirring bar.

5.2.2 Allow the suspension to stand for about 15 additional minutes with no agitation in order to allow most of the suspended particulates to settle out.

> Note: If the waste is hygroscopic and absorbs all the DIW, begin the test again using 20 g of waste and 40 ml of DIW.
>
> Note: If the supernatant is multiphasic, decant the oily phase and measure the pH of the aqueous phase.

5.2.3 Immerse the electrode (including the temperature correction probe) just far enough into the solution to establish good electrical contact. It should not come into contact with the waste material itself.

5.2.4 The electrodes should be thoroughly rinsed between samples.

5.2.5 Report the results as "waste pH measured in water @ _____ °C.

6. Reports

6.1 Data Packet Organization

6.1.1 The data package for this analysis consists of the data sheet and a quality control database (QC_DB) report form.

6.2 Quality Control Reports

6.2.1 All results for quality control tests are entered into the lab data base using the QC_DB program. The report form must be filled out in order to indicate that the duplicate QC had been performed and met the criteria specified in this document.

Appendix I

QC Summary Table

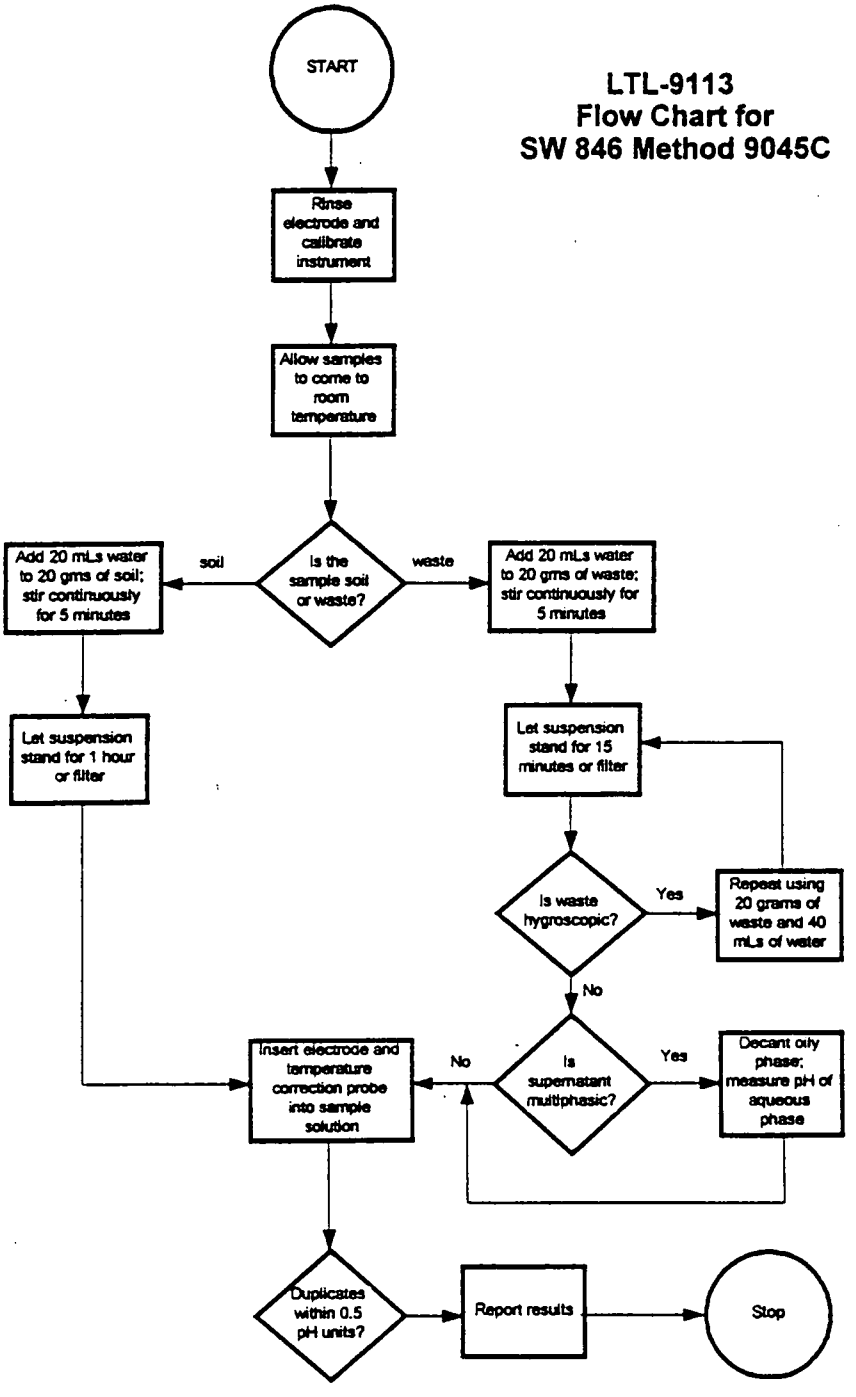
Laucks Testing Laboratories

Method SW 9045C QA Requirements and Corrective Actions

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Initial Calibration	Buffer pH meter to bracket sample values	Buffer pH meter between pH 7 & 4 or 10 before analyses (to bracket sample values)	Once before analyzing any samples or QC	None	None
Duplicate % Difference	N/A	within 0.5 pH units of one another	10% or once every 10 samples.	Correct problem Consult QC Officer	QC_DB report form with appropriate commentary

Appendix II

Flow Chart


LTL-9113
Flow Chart for
SW 846 Method 9045C
START
Rinse electrode and calibrate instrument
Allow samples to come to room temperature
Is the sample soil or waste?
soil
waste
Add 20 mLs water to 20 gms of soil; stir continuously for 5 minutes
Add 20 mLs water to 20 gms of waste; stir continuously for 5 minutes
Let suspension stand for 1 hour or filter
Let suspension stand for 15 minutes or filter
Is waste hygroscopic?
Yes
Repeat using 20 grams of waste and 40 mLs of water
No
Is supernatant multiphasic?
No
Yes
Decant oily phase; measure pH of aqueous phase
Insert electrode and temperature correction probe into sample solution
Duplicates within 0.5 pH units?
Report results
Stop

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-9110

Determination of Anions by Ion Chromatography by Method 300.0A or 9056

Revision history:

Number	Date
6	4/22/98
5	1/12/98
4	3/08/97
3	6/19/96
2	5/08/96
1.1 (LTL-0030)	5/31/90
1.0 (LTL-0030)	10/19/89

Written by:: Bill Lundberg Date: 4/22/98
Bill Lundberg, Inorganics Division Manager

Approved by: Harry Romberg Date: 4-22-98
Harry Romberg, QA Officer

Approved by: Kathy Kreps Date: 4-22-98
Kathy Kreps, Laboratory Director

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-9115

Title: **The Determination of Total Organic Carbon in Water Samples by SW846 9060.**

Revision history:

Number	Date
2	01/19/98
1.0	2/22/95

UNCONTROLLED

Written by: Bill Lundberg — Date: 1/29/98
Bill Lundberg, Inorganics Supervisor

Approved by: Harry Romberg — Date: 1-29-98
Harry Romberg, QA Officer

Approved by: Karen J Kotz — Date: 1/29/98
Karen Kotz, Laboratory Director

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APPENDIX II **18**

1. Introduction and Scope

1.1 Method Description

1.1.1 This method is used to determine the concentration of total organic carbon (TOC) in a water sample. The purgeable organic carbon (POC) is measured by sparging the acidified sample with oxygen for 3-5 minutes. The evolved gas is passed through a LiOH column to remove carbon dioxide formed from inorganic carbon (carbonates), combusted in a 950°C furnace, and measured using an IR detector. A system blank is subtracted from all POC readings before calculations are performed, this is to correct for any volatile carbon that may be present in the air at the time of analysis. Non-purgeable organic carbon (NPOC) is measured by injecting sparged, acidified sample into a chamber where a UV persulfate oxidation is performed. Carbon dioxide is evolved and measured in an IR detector. TOC is obtained by adding the POC and NPOC.

1.1.2 Method SW846 9060 states that all TOC analyses should be performed in quadruplicate. Laucks performs all TOCs in singlets, unless previous arrangements have been made with the client.

1.1.3 This procedure is applicable only to homogeneous samples which can be injected into the apparatus reproducibly by means of a microliter-type syringe. The openings of the syringe limit the maximum particle size which may be included in the sample..

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

1.2 Sample Collection, Sample Storage, Holding Times

1.2.1 It is s rongly recommended that samples be collected in glass containers preserved with 0.2% sulfuric acid. Samples collected in plastic run the potential risks of: A) an increased TOC value due to organic carbon leaching from the plastic into the sample and/or B) a decreased TOC value due to solvents in the sample becoming incorporated into the plastic container.

1.2.2 In accordance with SW846 plastic containers are permissible. Laucks will however make every effort to provide glass containers.

1.2.3 Upon receipt samples should be stored at 4 ± 2°C.

1.2.4 The maximum recommended holding time for waters is 28 days from the date of sample collection. If this holding time is exceeded, the analyst should notify the project manager so that they may inform the client.

1.3 Definition of Terms

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

1.3.2 Batch Identifier - A number given to each preparation or analysis group which uniquely identifies that batch. This number is generally an analysis number which is similar to the blank ID, only preceded by an "A" rather than a "B" for inorganic batches. The preparation batch IDs are discussed in other documentation. The batch identifier for the second set of TOC in water analyzed on January 19,1998 would be A011998TOCW02

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

1.3.4 CCB - Continuing Calibration Blank - This is the same acronym used in the CLP program. This is a blank which is analyzed immediately after the CCV (almost always after every 10 samples and at the end of the analytical run) during the analysis sequence to determine whether the instrument or system has maintained a stable baseline.

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

1.3.6 CLP - Contract Laboratory Program - The USEPA program that contracts with laboratories to provide laboratory services. The term has come to mean a much broader set of methods and deliverables. In context of this SOP, CLP means procedures or operations which are detailed in the CLP contract and which are extended to a broader working definition.

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

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

1.3.9 ICV - Initial calibration verification - This term is borrowed from the CLP protocol. It is a standard which is analyzed at the start of each analytical run that is compared to the initial multi-point calibration to determine whether the instrument calibration is accurate. For most inorganic methods, this verification standard is from a source different from that used to make the calibration standards.

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

1.3.11 SRM or LCS - Standard Reference Material or Laboratory Control Sample. This is a material of approximately the same matrix as the samples, containing a known and usually certified amount of target analyte and which is prepared and analyzed in the same manner as a typical sample. This sample is used to demonstrate that the analytical system is in control..

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

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

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

2. Equipment List and Standards

2.1 Instrument

2.1.1 TOC analyzer, Dohrman DC-80 or equivalent with POC furnace

2.1.2 Series of Unimetrics microliter syringes (1000, 200, and 40) or equivalent

2.2 Analytical balance: 0.1 mg sensitivity

2.3 Blender, Waring type, or equivalent.

2.4 Standards

2.4.1 NPOC potassium hydrogen phthalate (KHP) standards

2.4.1.1 Prepare a 4000 mg/l stock standard by dissolving 1700 mg ± 1 mg KHP in a 200 mL volumetric flask, dilute to the mark with DIW.

2.4.1.2 Prepare a 2000 mg/L intermediate standard by pipeting 50 ml of 4000 mg/L stock into a 100 ml volumetric flask, dilute to the mark with DIW.

2.4.1.3 Prepare 20 mg/L calibration standard by pipeting one ml of 2000 mg/L intermediate standard into a 100 ml volumetric flask, dilute to the mark with DIW

2.4.1.4 The 20 mg/l standard is prepared before each run. The 4000 and 2000 mg/l standards are to be made up monthly.

2.4.2 Preparation of POC Benzene standard

2.4.2.1 Prepare a stock solution of benzene in tetraglyme by adding about 25 ml of tetraglyme to a 50 ml volumetric flask. This should be done with a funnel in order to keep the neck of the flask dry. Cap the volumetric flask and weigh. Using a 5 ml glass syringe, draw up about 3 ml of benzene. Discharge the contents of the syringe into the volumetric flask just above the surface of the tetraglyme. Replace the cap and reweigh the flask. Fill to the mark with tetraglyme and mix well.

2.4.2.2 Calculate the concentration of the stock solution as follows:

$$\frac{\text{weight (mg) benzene}}{0.05} \times 0.923 = \text{mg/L C}$$

2.4.2.3 Prepare a POC intermediate standard by pipeting two mls of the stock solution into a 100 ml volumetric flask, dilute to mark with tetraglyme.

2.4.2.4 Prepare a working standard by filling a 100 ml volumetric flask to within 5 ml of the mark with DIW. Add 100 µl H_2SO_4, 100 µl antifoam, and one ml POC intermediate standard. Dilute to the mark with DIW. The working standard will be about 10 mg/L POC.

2.4.3 Preparation of glucose ICV solution

2.4.3.1 Prepare a 1000 mg/L stock solution by dissolving 250 mg ± 1 mg glucose in a 100 ml volumetric flask, dilute to the mark with DIW.

2.4.3.2 Prepare a 10 mg/L ICV solution by pipeting 1 ml of stock solution into a 100 ml volumetric flask, dilute to the mark with DIW.

3. Safety precautions and Waste Disposal

3.1 Safety Precautions

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

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

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

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

3.1.5 Breathing protection and gloves should be worn when handling benzene.

3.1.6 The combustion furnace is heated to approximately 950°C, care should be taken when working in proximity of the furnace.

3.1.7 Care should be taken when handling persulfate reagent due to the high Hg content and strong oxidizing capabilities.

3.2 Waste Disposal

3.2.1 All waste generated during this analysis is collected in plastic jugs. These are then collected by a waste recycling firm.

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

4. Calibration and Quality Control

4.1 Method Detection Limit Study

4.1.1 Prior to the analysis of any samples, it is necessary to establish method detection limits. This procedure is fully described in Laucks on MDL studies. Briefly, it involves the analysis of 7 replicate samples spiked at a concentration at 2-5 times the anticipated method detection limit. A Student's T-test is then applied to these measured values to calculate the MDL.

4.2 Initial Multi-Point Calibration

4.2.1 Analyze standard solutions using at least 3 different concentration levels. A 500ul injection ot the 20 mg/L standard is used to set the instrument's calibration, and a 1000ul injection of the 20 mg/L standard is used to define the upper range of useable data. A 250ul aliquot is used to demonstrate linearity.

4.2.2 Criteria

4.2.2.1 The correlation coefficient of the three points must be greater than .995.

4.2.3 Corrective action

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

4.3 Initial Calibration Verification

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

4.3.2 Criteria

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

4.3.3 Corrective action

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

4.4 Initial Calibration Blank

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

4.4.2 Corrective action

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

4.5 Continuing Calibration Verification (CCV) and Blank (CCB)

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

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

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

4.5.2 Corrective action

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

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

4.6 Matrix Spike

4.6.1 A sample is chosen at random from the samples to be analyzed, and an aliquot of spiking solution is added to this sample prior to preparation. The analyst should attempt to avoid selecting samples which are identified by the client as blanks. As the purpose of the matrix spike is to test the system under "typical" conditions, the analyst may also avoid selecting the most difficult sample of the batch for spiking. It is not always required that a matrix spike analysis be performed with each preparation/analysis batch, however, the minimum frequency for MS analysis is 1 each per 10 samples per matrix. This will be best accomplished by running one with every batch for many analyses. This matrix spike sample is used to evaluate the matrix effect of the sample upon recovery of the analytes. The recovery of spike analytes is calculated as follows:

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

where:

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

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

4.6.3 Corrective action

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

4.7 Matrix Spike Duplicate

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

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

where:

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

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

4.8 Sample Replicates

4.8.1 Criteria

4.8.1.1 The method requires that all samples be analyzed in quadruplicate. It then requires that the mean and the range be reported. Some clients may chose to have their samples analyzed at a lower frequency. If this is the case, then samples will be analyzed in duplicate with a minimum frequency of 1/20.. RPD values are calculated in a manner similar to MS/MSD RPDs:

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

where:

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

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

4.8.2 Corrective action

4.8.2.1 If a trend in out of control RPD values is observed, the methods used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable reproducibility. Generally, if recoveries are in control and no analyte of interest was detected in any of the samples, no immediate action will be taken on that sample set. If integrity of reported sample values is in doubt, re-analysis may be called for. Corrective actions should be discussed with the Quality Control Officer.

5. Operation procedures

5.1 Sample Analysis

5.1.1 Sample prep

5.1.2 Verify that the pH of the sample is <2.

5.1.3 If the sample contains a visible amount of suspended material, it should be homogenized by using the blender until it appears that all the sample reduction has taken place. If the sample still appears to be non-homogeneous, the analyst should disuss the situation with the project manager.

5.1.4 The POC portion of the analysis *must* be completed before homogenization, due to potential loss of volatile organic carbon.

5.1.5 Analysis sequence

NPOC Calibration standard (10 mg/L)
20 mg/L linearity check standard
5 mg/L linearity check standard
NPOC method blank
SRM
Matrix blank
POC method blank (3x)
System blank (3x)
POC standard
10 mg/L glucose ICV
ICB Blank (NPOC)
10 or fewer POC readings (may include QC)
10 mg/L NPOC standard (CCV)
Blank (NPOC, CCB)
Additional POC readings (10 maximum)

10 mg/L NPOC standard (CCV)
Blank (NPOC, CCB)

10 or fewer NPOC readings (may include QC)
10 mg/L NPOC standard (CCV)
Blank (NPOC, CCB)
Additional NPOC readings (10 maximum)
10 mg/L NPOC standard (CCV)
Blank (NPOC, CCB)

10 mg/L NPOC standard (CCV)
Blank (NPOC, CCB)

5.2 Instrumental Conditions

5.2.1 The instrument with POC furnace is assembled according to manufacturers instructions.

5.2.2 A gas flow of 200 ± 20 ml/min. oxygen is set for the furnace.

5.2.3 A reagent flow of 3.2 to 3.8 ml/min. is set for the recycle pump tube.

5.2.4 A reagent flow of 0.6 to 0.8 ml/min. is set for the fresh reagent pump tube.

5.3 Analytical Operation

5.3.1 Instrument startup

5.3.1.1 The furnace is turned on and allowed 90 min. to stabilize.

5.3.1.2 The reactor vessel is filled with reagent, pump and UV lamp are turned on, and the vessel is given at least 30 min. to stabilize.

5.3.1.3 Instrument is set for one ml sample size and for TOC mode.

5.3.1.4 When POC readings are being determined, the mode switch is set to POC.

5.3.2 POC determinations

5.3.2.1 POC readings must be made prior to NPOC reading due to potential loss of POC when opening the sample container.

5.3.2.2 With the instrument set to POC mode a one ml sample is injected into the POC sparger.

5.3.2.3 Push the start button, sample is sparged for 3.5 minutes, and the POC value is printed on the strip chart. If the value is higher than the highest standard, the sample must be diluted and reanalyzed

5.3.2.4 The determined system blank value is subtracted from the strip chart value before any calculations are done.

5.3.2.5 Proceed with the next POC reading or CCV.

5.3.3 NPOC determination

5.3.3.1 Sample is placed in a solo cup and sparged with the external sparger for 3.5 minutes.

5.3.3.2 With the instrument in TOC mode an appropriate amount of sample is injected into the instrument via the injection port, the NPOC value is then printed on the strip chart. If the value is higher than the highest standard, the sample must be diluted and reanalyzed

5.3.3.3 If NPOC is below one mg/L and less than one ml of sample was injected, rerun with a larger size aliquot.

5.3.3.4 If time out error occurs, wait for the detector to return to baseline and rerun sample using a smaller aliquot.

5.3.3.5 According to manufacturers instructions samples displaying a NPOC greater than 20 mg/L must be diluted and rerun. A 20 mg/L verification standard is run to demonstrate that the curve is linear to 20 mg/L.

5.4 Compound Quantification

5.4.1 TOC is determined by adding the POC and NPOC values together after they have been multiplied by their appropriate dilution factors.

6. Reports

6.1 Data Packet Organization

6.1.1 A data package will consist of the Excel spreadsheet used to quantify the TOC and a copy of the QC-DB report summary. If the client requests, the instrument output will also be provided.

6.2 Quality Control Reports

6.2.1 In addition to the sample results, all results for the quality control tests are entered into the lab data base using the QC_DB program. These results include those for the method blank analyses (both NPOC and POC method blanks are reported), matrix spike results, duplicate results, and standard reference material (SRM) reports. It is not necessary to include a copy of the information entered into the QC_DB program with each job. However, a copy of the QC_DB report form must be included in order to indicate that the QC was evaluated for analytical consistency.

6.3 Sample Result Reports

6.3.1 Data Qualifying Flags

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

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

6.4 Control Chart(s)

6.4.1 The recovery values for the LCS/SRM are plotted on control charts which may also be supplied to clients upon request.

Appendix I

QC Summary Table

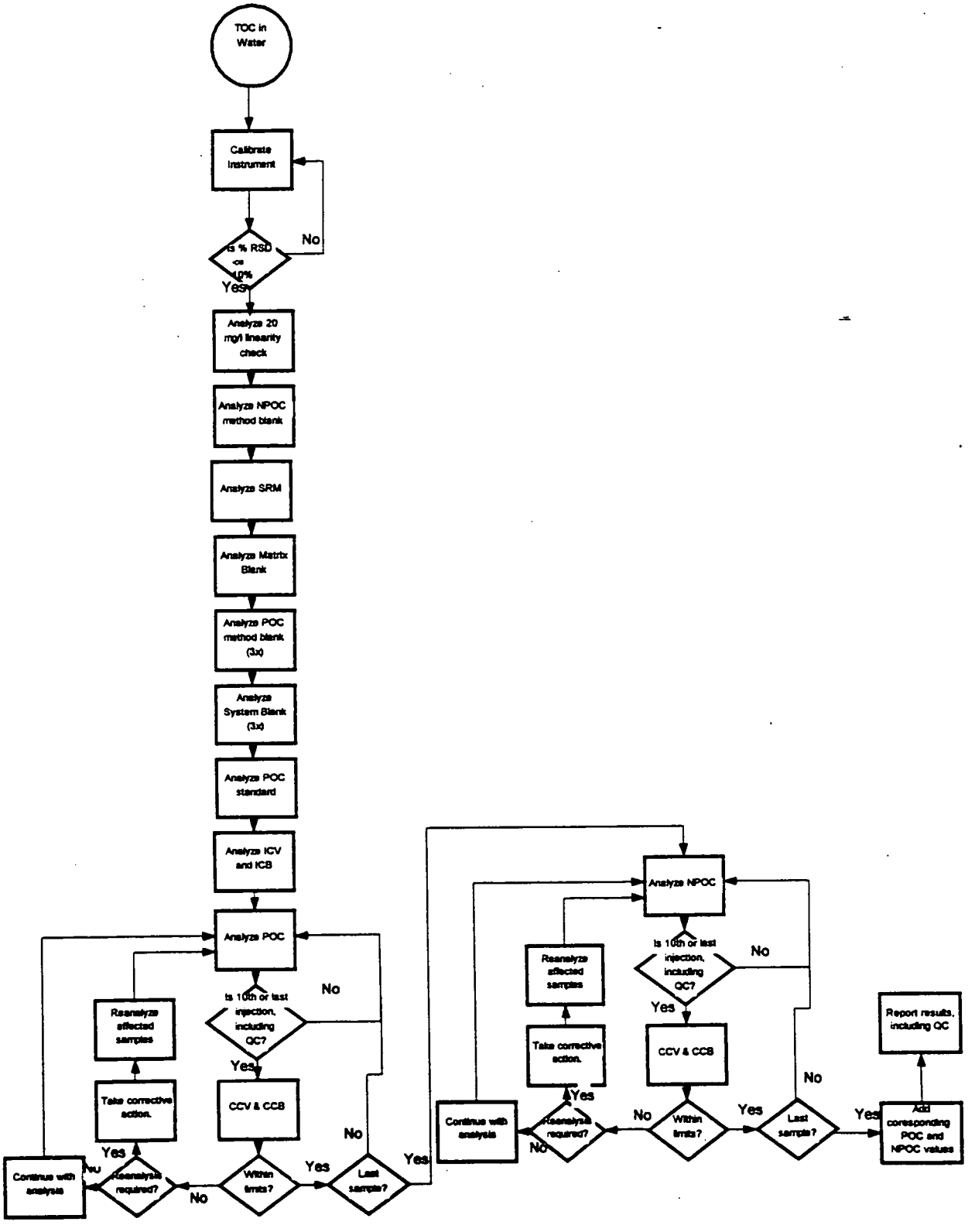
Laucks Testing Laboratories
Method _9060 QA Requirements and Corrective Actions

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Initial Calibration	Per Manufacturers Instructions	Per Manufacturers Instructions. See SOP section 4.2.	Once per run	Fix problem and re-calibrate	In raw data package
Initial Calibration Verification	Verify with an independently prepared check standard. No limits stated.	Verified with 10 mg/l glucose standard. Limits ± 10% (9.0 - 11.0)	Once per run	Reanalyze, remake solution, and/or recalibrate	In raw data package
Initial Calibration Blank	N/A	Must be less than reporting limit	Run after ICV	Reanalyze	In raw data package
Continuing Calibration Verification	Every 15 samples with an independently prepared standard, no limits	Verify with 10 mg/L standard; CCV must be within 10% of true value	After every 10 injections and at the end of the run.	Fix problem and re-calibrate; re-analyze samples which are not bracketed by valid CCVs	In raw data package
Continuing Calibration Blank	N/A	Must be less than reporting limit	Run after each CCV	Reanalyze	In raw data package
Linearity Check Standard	N/A	Verify with 20 mg/L standard; must be within 10% of true value	Once after NPOC calibration standard	Reanalyze, remake and/or recalibrate instrument	In raw data package

POC standard	N/A	Must meet current spike recovery limits	Once after POC method and system blanks	Remake standard	In raw data package
Method Blank	Once per sample batch; must be less than MDL	Must be less than reporting limit	One POC and one NPOC per batch	Reanalyze	In raw data package
Matrix Spike Recovery	Once every 10 samples; no criteria	Refer to current QC limits catalog and/or QC database	One MS/MSD pair per run or per 20, which ever is greater	Review data; consult QC officer; some conditions may require re-analysis	Narrative comment in report and raw data package
MS/MSD RPD	N/A	Refer to current QC limits catalog and/or QC database	One MS/MSD pair per run or per 20, which ever is greater	Review data; consult QC officer; some conditions may require re-analysis	Narrative comment in report and raw data package
Duplicate % Difference	N/A	Refer to current QC limits catalog and/or QC database	Once every 20 samples	Review data; consult QC officer; some conditions may require re-analysis	Narrative comment in report and raw data package
Standard Reference Material (SRM) Recovery	N/A	Values are supplied by manufacturer	Once every 20 samples	Review data; consult QC officer	Narrative comment in report and raw data package

Appendix II

Flow Chart


TOC in Water
Calibrate Instrument
Is % RSD <= 10%
No
Yes
Analyze 20 mg/l linearity check
Analyze NPOC method blank
Analyze SRM
Analyze Matrix Blank
Analyze POC method blank (3x)
Analyze System Blank (3x)
Analyze POC standard
Analyze ICV and ICB
Analyze POC
Is 10th or last injection, including QC?
No
Yes
CCV & CCB
Within limits?
Yes
No
Last sample?
Yes
No
Reanalyze affected samples
Take corrective action.
Reanalysis required?
Yes
No
Continue with analysis
Analyze NPOC
Is 10th or last injection, including QC?
No
Yes
CCV & CCB
Within limits?
No
Yes
Last sample?
No
Yes
Reanalyze affected samples
Take corrective action.
Reanalysis required?
Yes
No
Continue with analysis
Report results, including QC
Add corresponding POC and NPOC values

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-9202

Title: **Total Suspended Solids (Non-Filterable Residue) Using EPA Method 160.2 / Standard Methods 2540D and Total Volatile Suspended Solids Using Standard Methods 2540 E.**

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1. Introduction and Scope

1.1 Method Description

1.1.1 Scope and Application

1.1.1.1 This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes. The practical range of the determination is 4 mg/l to 20,000 mg/l. The detection limit, however, is generally reported as 2 mg/l unless a sample volume greater than 100 mls is used.

1.1.1.2 A well-mixed sample is filtered through a standard glass fiber filter. The filter and residue are dried to constant weight at 103°C-105°C.

1.1.1.3 The filtrate from this method may be used for Total Dissolved Solids (Filterable Residue).

1.1.1.4 If volatile suspended solids are to be determined, the filter and residue are then ignited at 550°C and the loss on ignition determined.

1.1.2 Interferences

1.1.2.1 Samples high in dissolved solids (filterable residue), such as saline waters, brines and some wastes, may be subject to positive interferences due to soluble material which has not been adequately washed from the filter. Care must be taken to ensure that an appropriate filtering apparatus has been selected and the filter adequately washed in order to minimize this possibility.

1.1.2.2 Samples which are very high in suspended material or which have certain particle sizes may plug the filter causing difficulty in filtering. It may be necessary to reduce sample size in order to reduce this tendency.

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

1.2 Sample Collection, Sample Storage, Holding Times

1.2.1.1 No preservation of the sample is necessary. Refrigeration or icing to 4°C, to minimize microbiological decomposition of solids, is required. Analysis should begin as soon as possible and should in no case exceed 7 days from the date of collection. All exceptions, whether due to laboratory or client cause, must be recorded on a Holding Time Violation Report (HTVR).

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.

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

MDL - Method detection limit - The lowest concentration a sample which will yield a positive result that is greater zero at a known level of confidence. MDLs are empirically determined by Laucks, although there is no known way to determine the MDL for the TSS or TVSS analyses.

SRM or LCS - Standard Reference Material or Laboratory Control Sample. This is a material of approximately the same matrix as the samples, containing a known and usually certified amount of target analyte and which is prepared and analyzed in the same manner as a typical sample. This sample is used to demonstrate that the analytical system is in control. It may be considered to be a blank spike for most inorganic analyses and is preferred over artificially spiking blank materials. This type of sample is rarely analyzed at Laucks for TSS and there is no known source of TVSS material.

Sequence - A set of samples analyzed in a chronologically continuous group.

Total Suspended Solids (TSS) (nonfilterable residue)- Those solids which are retained on a glass fiber filter of the appropriate retentive ability and dried to constant weight at 103-105C.

Total Volatile Suspended Solids (TVSS) - Suspended solids which are volatile (burn off) at a temperature of 550°C.

2. Equipment List

Glass fiber filter discs, 4.7 cm without organic binder, Gelman A/E or equivalent. Laucks currently uses Pro Weigh 47 mm glass fiber filters from Environmental Express which are designed for TSS analysis. They are pre-washed and pre-weighed and require no additional preparation.

Membrane filter funnel with the capability of adequately supporting 4.7 cm. filters

Suction flask

Drying oven, set at 103°C-105°C.

Muffle furnace set at 550°C ± 50°C

Desiccator charged with active silica gel desiccant

Analytical balance, capable of weighing to 0.1 mg.

3. Safety precautions and Waste Disposal

3.1 Safety Precautions

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

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

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

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

3.1.5 Routine precautions include an awareness of elevated temperatures of both the oven and any samples which have recently been removed from the drying oven or especially the muffle furnace. The temperatures involved in this analysis can cause severe burns if adequate care is not taken.

3.2 Waste Disposal

3.2.1 No waste should be generated from this procedure. Sample residues may be washed out and discarded down the sink.

4. Quality Control

4.1 Method Blanks

4.1.1 Criteria:

4.1.1.1 Method blanks are used to verify contamination free reagents and apparatus. They are prepared with every set of samples prepared at the same time or at least one blank every 20 samples which ever is more frequent. The method blank is prepared by pouring the same amount of deionized water through a filter as one would a typical sample (generally 100 mls.). Any TSS above the detection limit is reported. Method blank control limits are such that contamination should not exceed twice the detection limit. If 100 ml samples are used, Laucks generally considers the detection limit to be 2 mg/L with method blank control limits of 4 mg/L.

4.1.1.2 In making a determination of whether or not the analysis is in control, the analyst should actually be normalizing the blank to whatever volume was used for any sample. It is assumed that any blank is due to washings from the filter and apparatus and is actually independent of sample volume. In other words, the control limit for this analysis is actually 0.4 mg. If blank contamination exceeds that value, the TSS (or TVSS) of samples must be ten times that weight or corrective action should be taken.

4.1.1.3 If TVSS is being determined, the filters are processed in the same manner only are also heated to 550°C. Otherwise, the detection limits, criteria, and corrective actions are the same as for TSS.

4.1.2 Corrective action

4.1.2.1 Corrective action may necessitate re-preparation and re-analysis of the sample set. For example if TSS or TVSS were found in the blank but not in any of the associated samples, then the sample group may not require re-analysis. In addition, if sample levels exceed 10 times the blank, the level of contamination may be considered insignificant. In any case, if re-analysis is not being undertaken, the analyst must first discuss the issue with the Quality Control Officer. In all cases where blank contamination exceeds the control limit, a narrative comment must be made which documents the corrective actions taken.

4.2 SRM or LCS

4.2.1 Criteria

4.2.1.1 Analysis of a reference material is not normally required for TSS analysis and no known material is available for TVSS. An SRM/LCS analysis will generally be analyzed only if specifically required for a project. If not otherwise specified in that contract, it would typically

be analyzed at a frequency of once per 20 samples. Vendor specified control limits would be used for any such material.

4.2.2 Corrective Action

4.2.2.1 Re-analysis of all associated samples may be required if this sample exceeds it's limits. The QC Officer should be consulted for any other corrective actions and all instances of out-of-control events and any actions taken must be documented in the QC narrative of the report.

4.3 Sample Duplicate

4.3.1 Criteria
At least one duplicate sample per 10 samples is required. RPD values are calculated as follows:

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

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

4.3.1.1 The RPD control limits are detailed in the Quality Control Database (QC_DB) and will change from time to time. For samples with values which are less than 5 times the detection limit, the control limit is equal to 5 times the detection limit. For values greater than 5 times the detection limit, the control limit is a calculated percent RPD.

4.3.2 Corrective action

4.3.2.1 If a trend in out of control RPD values is observed, the methods used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable reproducibility. If integrity of reported sample values is in doubt, re-analysis of all associated samples may be called for. Corrective actions should be discussed with the Quality Control Officer.

5. Operation procedures

5.1 Sample Analysis

5.1.1 Analysis sequence

Method Blank
SRM or LCS (if required)
up to 20 samples plus duplicates

5.1.2 Analytical Operation

5.1.2.1 Drying, ashing or cooling times in this section are those typically used in the laboratory and are not specifically required by the method. The method requires a minimum of 1 hour drying or ashing times and analysis to constant weight. This may be achieved through either shorter or longer time periods than those specified in this SOP. Experience has also shown that the beakers or other weighed containers must be cooled to room temperature for the weights to be stable and accurate. As long as proper and complete cooling has been allowed to occur in an environment where the dried or ashed sample cannot re-absorb water or be otherwise contaminated, these times are not specified in the published method and are thus also flexible.

5.1.2.2 Preparation of glass fiber filter disc if Environmental Express Pro Weigh filters are being used: Remove the filter from it's aluminum weighing dish and place the disc on the membrane filter apparatus with the wrinkled side up. While vacuum is applied, rinse the disc with a small amount of deionized water in order to seat the disk in the filter device.

5.1.2.3 Preparation of glass fiber filter disc if pre-washed and pre-weighed filters are NOT being used: Place the glass fiber filter on the membrane filter apparatus (if 4.7 mm filters are being used) or insert into bottom of a suitable Gooch crucible (if 2.4 cm filters are being used) with the wrinkled surface up. While vacuum is applied, wash the disc with three successive 20 ml volumes of distilled water. Remove all traces of water by continuing to apply vacuum after water has passed through. Remove filter from membrane filter apparatus (if 4.7 mm filter) or both crucible and filter if Gooch crucible is used, and dry in an oven at 103°C-105°C for one hour or at 550°C ± 50°C if TVSS is to be determined. Remove to desiccator and store until needed. Repeat the drying cycle until a constant weight is obtained (weight loss is less than 0.5 mg). Weigh immediately before use. After weighing, handle the filter or crucible/filter with forceps or tongs only. As in the procedure for the pre-weighed filters above, wet the filter slightly before use in order to properly seat it in the filtration apparatus.

5.1.2.4 Assemble the filtering apparatus and begin suction, taking care to have wet the filters as described in either procedure above. Shake the sample vigorously and rapidly transfer 100 ml to the funnel by means of a 100 ml graduated cylinder. If this volume takes longer than 5-10

minutes to pass through the filter, sample volume must be reduced such that the filtration time will not be exceeded. If that volume is less than or equal to 10 mls, it should be dispensed with a 10 ml Mohr (graduated glass) pipet which has a wide enough tip opening so as not to inhibit the passing of solid pieces of material.

5.1.2.5 Filter the sample through the glass fiber filter, rinse the graduate and filter with three successive 10 ml portions of deionized water, allowing the rinsate to pass completely through the filter between washings, and continue to apply vacuum for about 3 minutes after filtration is complete to remove as much water as possible.

5.1.2.6 If the sample contains large pieces of material which make it difficult or impossible to achieve a representative, homogeneous sample, it may be necessary to thoroughly mix the sample in a blender prior to filtration. If this is done, however, care should be taken to assure that air bubbles aren't entrained in the measured sample volume to the extent that it could affect the actual volume dispensed. In other words, let the bubbles and subsequent foaming subside before dispensing the sample.

5.1.2.7 Carefully remove the filter from the filtration device, taking care not to leave pieces of filter on the support apparatus, and place it in the aluminum weighing dish appropriate to that filter and sample.

5.1.2.8 Dry the filter overnight at 103°C-105°C.

5.1.2.9 Remove the filter and aluminum dish from the oven and place in a desiccator to cool. The samples MUST be cooled prior to weighing or the apparent weight will be affected.

5.1.2.10 Repeat the cycle of drying, desiccating, cooling and weighing until the filter/dish attain a constant weight, changing by no more than 0.5 mg.

5.1.2.11 Record the final weight for calculation of TSS.

5.1.2.12 If TVSS is to be determined, repeat step 5.1.2.9 using a muffle furnace pre-heated to 550°C ± 50°C for 90 minutes. Record the final weight for calculation of TVSS.

5.2 Quantification

5.2.1 Residue concentrations are calculated using the following equations:

$$TSS = \frac{(W_f - tare) \times 1000}{V_i}$$

$$TVSS = \frac{W_f - W_a \times 1000}{V_i}$$

where:

V_i = volume of sample used in mls
W_f = weight of dried residue & filter
W_a = weight of ignited residue (after ignition @ 550°C) & filter in mg
tare = tare weight (weight in mg of filter before filtration of sample)

5.2.2 TSS and TVSS are generally reported on a mg/L as received basis.

6. Reports

6.1 Data Packet Organization

6.1.1 The data package for this analysis consists of the data sheet and a quality control database (QC_DB) report form.

6.2 Quality Control Reports

6.2.1 All results for quality control tests are entered into the lab data base using the QC_DB program. Printouts of all data entered need not be included in the package. However, all must be referenced on the report form. This includes the blank and duplicate results and any other QC which might have been analyzed by special request (such as an SRM/LCS).

6.3 Sample Result Reports

6.3.1 Data Qualifying Flags

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

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

Appendix I

QC Summary Table

Laucks Testing Laboratories

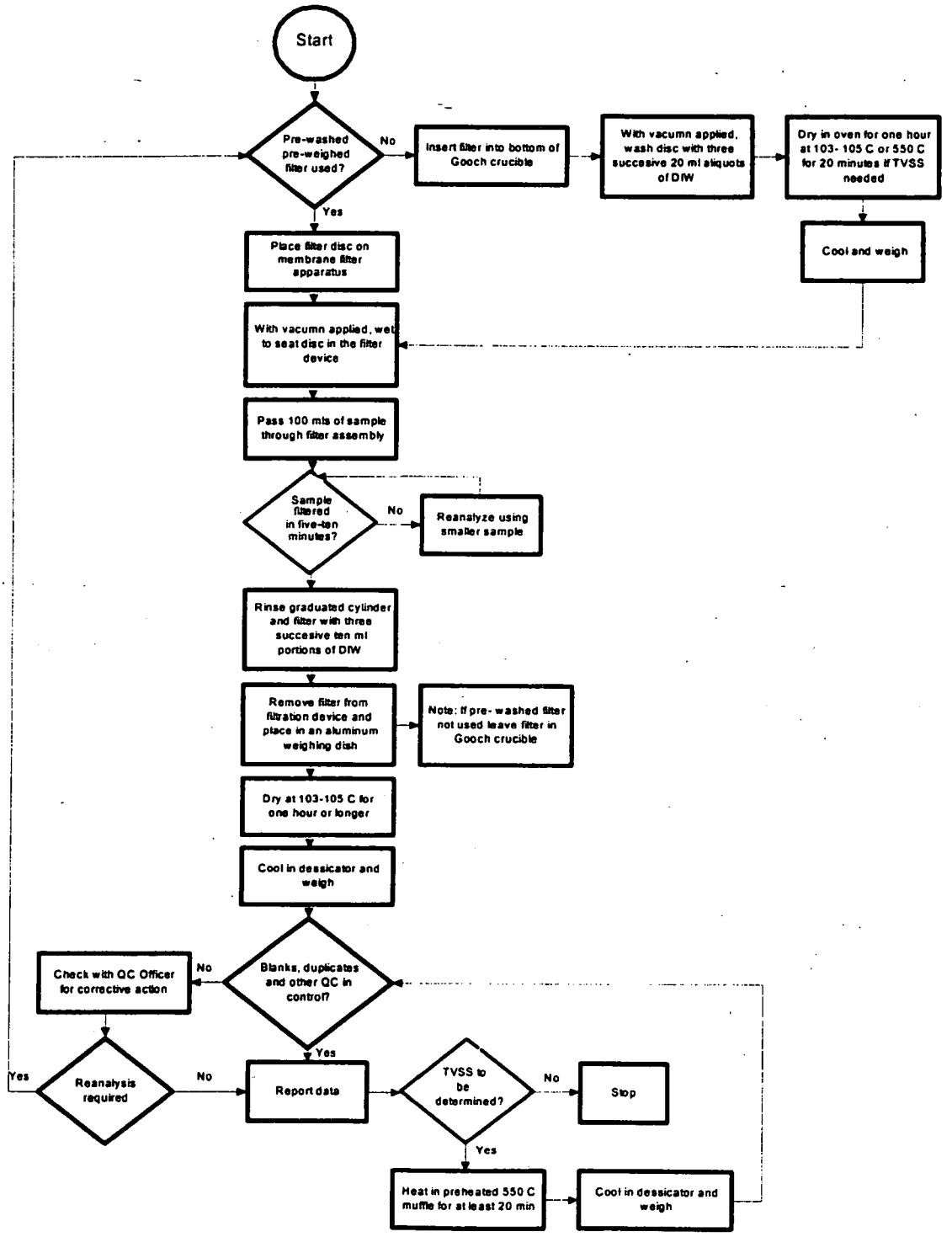
Method EPA 160.2 / SM 2540D & E QC Requirements and Corrective Actions

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Method Blank	None	<4 mg/L or no more than twice the detection limit	5% frequency (1 per 20 samples)	Re-analyze all samples <10x the actual weight of the solids. Consult QC Officer for any other actions	QC_DB report form with appropriate commentary
Duplicate % Difference	None	See current control limits catalog or QC_DB database	10% frequency (1 per 10 samples)	Discuss with QC Officer. If impact appears serious, may need to re-analyze samples	QC_DB report form with appropriate commentary
Standard Reference Material (SRM) Recovery	None	Within vendor supplied limits or 90%-110% recovery	If required, at frequency specified, or 5% if not specified	Discuss with QC Officer. If impact appears serious, may need to re-analyze samples	QC_DB report form with appropriate commentary

Appendix II

Flow Chart

Total Suspended Solids (TSS) and Total Volatile Suspended Solids (TVSS)
EPA 160.2 and/or SM 2540D&E



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Document Control and Tracking

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March 29, 2000

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Date: _____

DRAFT ONLY

1.0 PURPOSE: This document outlines the procedures for controlling and tracking all documents originating from and to the Explosive Sciences Branch. It ensures that all technical documents used by the ESB are complete, accurate, current and distributed to the appropriate personnel, and can be readily retrieved.

2.0 SCOPE: These procedures describe the method for assigning document control numbers to documents and for tracking any incoming documents received by the Explosive Sciences Branch. This applies to the approval, issue, storage, and revision of all technical documents, customer reports, and equipment operating procedures.

3.0 PROCEDURE:

3.1. Document Control for Procedure Documents

3.1.1. The document must be routed and approved.

3.1.2. Once the document has been approved, the preparer must log-in the document into the file server in order to provide a document control number. The following information must be provided to the file server to ensure proper document control: Title, Date, and Author. This may be performed by the preparer who has had the proper training in assigning document control numbers.

3.1.3. All documents should be given to the document control custodian via electronic means (on disk or e-mailed). If that is not possible, then a hard copy must be sent. The resulting document will be permanently stored in the document control system, while the hard copy will be stored within the Explosive Sciences Branch building.

3.1.4. The document control custodian shall maintain a master list of all controlled documents and their revision status and shall be able to generate the list upon request.

3.1.5. Personnel requesting a hard copy may download the document from the file server.

3.2. Changes/Revisions for Procedure Documents

3.2.1. The original preparer of the controlled document, when possible and practical, shall review and approve changes/revisions, and shall note the changes in the document or on an attachment before routing for approval.

3.2.2. When final approval is received, the preparer shall log-in the revised document on the file server and send an electronic copy to the document custodian. The revision shall be noted and a new document control number will be assigned to reflect the revision.

3.3. Document Control for Customer Reports

3.3.1. The control number must be obtained from the Word Processing Log binder, located at the front of the office. The following information must be written in the log: originator's initials and report subject.

3.3.2. All customer reports must have supervisor's verbal approval before submission to the document custodian, via floppy disk and hard copy.

3.3.3. The final customer report, signed by the supervisor, will be sent back to the preparer for initials.

4.0 RESPONSIBILITY:

4.1. DEPARTMENT MANAGER

4.1.1. Ensure current and appropriate documents are available at all locations where operations take place.

4.1.2. Establish documentation repositories, as required.

4.1.3. Appoint a document custodian and alternate.

4.1.4. Ensure this policy is available and followed by all Department Personnel.

DRAFT ONLY

4.2. DOCUMENT RECIPIENT/USERS

4.2.1. Inform custodian of any changes in organization, code, or location.

4.2.2. Ensure current and appropriate documents are available at all locations where operations take place.

4.3. DOCUMENT CUSTODIAN

4.3.1. Maintain and control documents submitted by department personnel.

4.3.2. Maintain a master list of all documents controlled by the document control system.

4.3.3. Introduce new documents into the system.

4.3.4. Issue controlled copies of documents.

4.4. PREPARERS

4.4.1. Prepare and maintain documents in accordance with Branch guidelines and this instruction.

4.4.2. Review and approve changes/revisions to documents originated.

4.4.3. State nature of changes on documents or on attachment.

4.4.4. Ensure that review copies of all new and revised documents are routed to pertinent personnel within and outside the Department.

4.4.5. Prepare and route electronic copies for final approval after initial routing for review/comments has bee completed.

4.4.6. Ensure that the document control custodian receives notification of final approvals.

4.4.7. Assign a document control number by logging into the document control system on the file server.

5.0 REFERENCE DOCUMENT:

CR-405-3001A; Directive for Control of Technical Documents and Engineering Drawings.

CR4052-PD-002
Revision: Original

Elements and Format of Procedure Documents

Explosive Sciences Branch

Naval Surface Warfare Center Crane

300 Highway 361

Crane, IN 47522

March 29, 2000

Approvals:

Name: Nancey Maegerlein
Title: Explosive Sciences Branch Manager
Signature: _____
Date: _____

Name: Jacquelyn Karty
Title: Chemist
Signature: _____
Date: _____

DRAFT ONLY

1.0 PURPOSE: This document describes the elements and format required for an approved procedure document. It is designed to help in the preparation and review of a procedure document. The terms "shall" and "must" are used when the element mentioned is required. The term "should" indicates that the element mentioned is recommended, while the term "may" indicates when the element is optional.

2.0 SCOPE: The procedure document is intended to be specific to the Explosive Sciences Branch. The documents are intended for the sole use of the ESB and may not be specifically applicable to other activities of other organizations.

3.0 GENERAL FORMAT:

3.1. Title Page
The first page of each procedure document should be a title page having the following information: a title that clearly identifies the activity or procedure, the name of the applicable agency/group, and the date and signatures of those individuals who prepared and approved the document.

3.2. Table of Contents
A Table of Contents is needed only if the procedure document is longer than ten pages.

3.3. Control Documentation
Each page of the procedure document should have control documentation. The information should be in the upper right a hand corner, containing the following information: document control number, revision number, date, and page number.

3.4 Text
A well-written procedure document has three basic sections: procedural, QA/QC, and reference. The text of the document should be clearly worded so as to be readily understandable by a person knowledgeable with the general concept of the procedure. The procedures should be written in a

step-by-step format that clearly describes the steps in chronological order.

4.0 SUGGESTED FORMAT FOR A TECHNICAL PROCEDURE DOCUMENT

4.1. Procedural Section

After the title page, the following are topics that may be appropriate for inclusion in a procedure document:

a) Scope and Applicability,
b) Summary of Method,
c) Definitions,
d) Health and Safety Warnings,
e) Cautions,
f) Interferences,
g) Personnel Qualifications,
h) Apparatus and Materials,
i) Instrument or Method Calibration,
j) Sample Collection,
k) Handling and Preservation,
l) Sample Preparation and Analysis,
m) Troubleshooting,
n) Data Acquisition, Calculations, and Data Reduction,
o) Computer Hardware and Software, and
p) Data Management and Records Management.

4.2. Quality Control and Quality Assurance Section

Specify and describe any QA procedures that are integral parts of the procedure. These may include performance evaluations, outside audits or reviews.

4.3. Reference Section

Documents or procedures that relate to the procedure document should be referenced (including version). Fully cite all references noted in the body of the SOP and attach any that are not readily available.

5.0 SUGGESTED FORMAT FOR AN ADMINISTRATIVE PROCEDURE DOCUMENT

5.1 Procedural Section

The following are topics that may be appropriate for inclusion in an administrative procedure document:

a) title,
b) Purpose
c) Applicability
d) Summary of Procedure
e) Definitions
f) Personnel Qualifications, and
g) Procedure

5.2 Quality Control and Quality Assurance Section
Describe any control steps and provisions for review or oversight prior to acceptance of the procedure document.

5.3 Reference Section
Cite all references noted in the body of the procedure document. A copy of any cited references not readily available should be attached to the document procedure.

6.0 REFERENCE DOCUMENTS
EPA/600/R-96/027; Guidance for the Preparation of Standard Operating Procedures for Quality-Related Documents (November 1995).

Internal Sample Chain of Custody

Explosive Sciences Branch

Naval Surface Warfare Center Crane

300 Highway 361

Crane, IN 47522

April 1, 2000

Approvals:

Name: Nancey Maegerlein_____
Title: Explosive Sciences Branch Manager
Signature: _____
Date: _____

Name: Jacquelyn Karty _____
Title: Chemist
Signature: _____
Date: _____

DRAFT ONLY

1.0 Purpose: Sample custody procedures are necessary to provide and maintain data validity and control. Samples must be under the control of the laboratory at all times after their transfer from the sample courier and during sample analysis. A chain-of-custody program provides legal support to the analytical results and tracking for each sample that is brought into the laboratory and during sample analysis.

Proper sample tracking is accomplished with a Chain-of-Custody (CoC) form and an Internal Chain-of-Custody (ICoC) form. The CoC form is generated in the field and used to record a sample's identity. The CoC shall accompany samples from the time of sampling until the time they are delivered and logged into the Explosive Sciences Branch (ESB) electronic inert sample log. The ICoC is used to record and track a sample from delivery at the laboratory through preparation, analysis, and disposal.

2.0 Scope: This procedure describes the method for completing, maintaining, and storing the ICoC for soil and water samples received by the ESB.

3.0 Receipt of Samples

All laboratory personnel that perform environmental sample analysis can accept samples delivered to the laboratory. Upon the receipt of the sample shipment and CoC forms, the receiving laboratory personnel will complete the following steps:

3.1 Inspect all shipping containers for signs of tampering (i.e. broken seals and/or damage to the shipping containers).

3.2 Inspect sample CoC documentation to ensure the following information is documented:

- 3.2.1 Customer's name and address
- 3.2.2 Customer's point of contact and phone number
- 3.2.3 Signature of sampler(s)
- 3.2.4 Sample ID, preservative used, extraction hold time, sampling date, time of collection, matrix type, container type, sample type, and the requested analysis.

3.2.5 Signatures, date, and time of individuals involved in the sample transfer (i.e. relinquishing and accepting samples)

3.3 Any samples which do not meet this inspection will be rejected and/or the discrepancies noted on the CoC form and in the ESB electronic sample log. The client's point of contact will be immediately notified of the discrepancies.

3.4 Samples received will then be logged into the laboratory inert electronic sample log that resides on the ESB file server, thus allowing limited and secure access to the sample log.

3.4.1 Open the file Y:/Code4076/Samp_log/Inert00.xls (Figure 1)
3.4.2 Enter the name of the person receiving the sample, the date the sample was received, the client, sample identification and description, analysis requested, laboratory sample identification number, and the analyst assigned to the analysis.
3.4.3 Record laboratory sample identification number for sample(s).
3.4.4 Save the file

3.5 Identify the sample identification on the CoC with the corresponding laboratory tracking sample number assigned in the inert electronic sample data log
3.6 Write the laboratory sample identification number on the sample container with permanent marker.
3.7 Place sample into the designated sample refrigerator.
3.8 File the original CoC form. CoC forms are kept on file at the laboratory for five years.

4.0 Internal Chain-of-Custody Form

After samples have been received and entered into the laboratory inert electronic sample log, an Internal Chain-of-Custody form must be filled out. This form will be kept near the place of storage for easy access during sample preparation and analysis. The following information must be filled out:

4.1 Project Name, job order number and charge number (assigned by financial department), project leader,

date ICoC log sheet started, customer, customer point of contact (POC), customer address and phone number (see Figure 2).

4.2 Each time a sample is place in or removed from the storage location the sample identification number, date of movement, time of movement, who removed it, and the reason for removal must be entered on the ICoC.

4.3 When the ICoC sheet is full, the page number is filled out, a new sheet is filled out with the exact same information as listed in 4.1, and the full sheet is filed with the CoC forms.

Figure 1: Laboratory Inert Electronic Sample Log

ESB Sample #	Date Received	Logged By	Sample ID/Description	Customer Contact	Test Requirements	Assigned To	Comments

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Figure 2: Internal Chain-of-Custody Form

Internal Sample Chain-of-Custody Record
300 Highway 361, Crane, IN 47522 Phone (812)854-5517 Fax: (812)854-5054

Project Name:
Job Order Number:
Charge Number:

Project Leader:
Date:
Page: of

Customer:
Customer POC:
Address & Phone #:

Sample Identification Number	Date	Time	Reason

DRAFT ONLY

CR4052-PD-004
Revision: Original

Glass Cleaning Procedure

Explosive Sciences Branch

Naval Surface Warfare Center Crane

300 Highway 361

Crane, IN 47522

March 29, 2000

Approvals:

Name: Nancey Maegerlein
Title: Explosive Sciences Branch Manager
Signature: _____
Date: _____

Name: Jacquelyn Karty
Title: Chemist
Signature: _____
Date: _____

DRAFT ONLY

1.0 PURPOSE: This document provides the proper cleaning procedure of all glassware in the ESB.

2.0 SCOPE: These procedures describe the method for removing surface residuals, washing the glassware, and drying of the glassware.

3.0 PROCEDURE:

3.1. Removing Surface Residuals
Any tape, label or marker stains must be removed prior to placing in the dishwasher. Tape and label may be removed with a knife, while marker stains can be removed with acetone.

3.2. Washing the Glassware
All glassware is placed onto steel or plastic-coated racks. Soap is added to the dishwasher and the door is brought down. The temperature of the dishwasher must be brought to 95 °C before the dishwasher may be started.

3.3. Drying the Glassware
Once the glassware has been washed, then the racks are placed in the Precision Mechanical Convection Oven. They are dried in the oven for approximately 60 minutes.

3.4. Rinsing the Glassware Prior to Use
All glassware is rinsed three times with the solvent to be used in the analysis.

DRAFT ONLY

Analytical Balances

Explosive Sciences Branch

Naval Surface Warfare Center Crane

300 Highway 361

Crane, IN 47522

April 1, 2000

Approvals:

Name: Nancey Maegerlein_
Title: Explosive Sciences Branch Manager
Signature:_____
Date: _____

Name: Jacquelyn Karty_
Title: Chemist_
Signature:_____
Date: _____

DRAFT

1.0 PURPOSE: This document outlines the procedures followed at Crane Division, Naval Surface Warfare Center (NAVSURFWARCENDIV Crane) for calibration of electronic analytical balances owned by the Explosive Sciences Branch (ESB).

2.0 SCOPE: This document references the NAVSURFWARCENDIVCRANE Instruction 4855.20 Calibration Program and describes internal ESB procedures for confirming calibration.

3.0 NAVSURFWARCENDIVCRANE Instruction 4855.20

NAVSURFWARCENDIVCRANE Instruction 4855.20 delineates policy, responsibilities, and procedures for the calibration program at NAVSURFWARCENDIV Crane. All analytical balances owned by the ESB are covered by this instruction.

3.1 The Calibration Laboratory and Repair Branch (Code 6034) provides calibration services to the ESB. Code 6034 calibrates and certifies each balance owned by the ESB every 2 to 3 years (depending on balance make and model).

3.2 Explosive Sciences Branch Internal Calibration Confirmation

Prior to use, any analytical balance calibration is confirmed using the following procedure:

3.2.1 Ensure the balance sample pan is clean. Tare the balance.
3.2.2 Place a 100 gram Navy calibration weight on the pan. Note final weight.
3.2.3 Tare balance and place a 10 gram Navy calibration weight on the pan. Note final weight.
3.2.4 Tare balance and place a 1 gram Navy calibration weight on the pan. Note final weight.
3.2.5 Code 6034 calibration is confirmed if the weights indicated by the balance are within ±0.005 grams of the Navy calibration weights listed in Sections 4.2, 4.3, and 4.4.
3.2.6 Code 6034 calibration is invalid if the weights indicated by the balance are outside ±0.005 grams of the Navy calibration weights listed in Sections 4.2, 4.3, and 4.4.

3.2.7 If the electronic analytical balance calibration is determined to be invalid, Code 6034 is contacted to re-certify the balance calibration.

3.2.8 If the electronic analytical balance is determined to be in need of repair, the manufacturer is contacted and arrangements are made for repair and re-calibration.

Refrigerator, Freezer, and Oven

Thermometer Calibration, Monitoring, and Maintenance

Explosive Sciences Branch

Naval Surface Warfare Center Crane

300 Highway 361

Crane, IN 47522

March 29, 2000

Approvals:

Name: Nancey Maegerlein
Title: Explosive Sciences Branch Manager
Signature: _____
Date: _____

Name: Jacquelyn Karty
Title: Chemist
Signature: _____
Date: _____

DRAFT

1.0 PURPOSE: This document outlines the procedures followed at Crane Division, Naval Surface Warfare Center (NAVSURFWARCENDIV Crane) for thermometer calibration, monitoring, and maintenance of refrigerators, freezers, and ovens.

2.0 SCOPE: This document references the NAVSURFWARCENDIV Crane Instruction 4855.20 Calibration Program and describes internal ESB procedures for monitoring and maintenance of refrigerators, freezers, and ovens.

3.0 NAVSURWARCENDIVCRANE Instruction 4855.20

3.1. NAVSURWARCENDIVCRANE Instruction 4855.20 delineates policy, responsibilities, and procedures for the calibration program at NAVSURWARCENDIV Crane. While the refrigerators, freezers, and ovens used by the Explosive Sciences Branch (ESB) are not directly covered by this instruction, the thermometers used to monitor the refrigerators, freezers, and ovens are covered by this instruction.

3.2. The Calibration Laboratory and Repair Branch (Code 6034) provides calibration services to the ESB. Code 6034 certifies thermometers for use in the ESB.

4.0 EXPLOSIVE SCIENCES BRANCH INTERNAL MONITORING PROGRAM

4.1. Refrigerators and Freezers

Refrigerators and Freezers are monitored according to the following procedure:

4.1.1 Each day a thermometer is placed in the refrigerator or freezer. The thermometer is left in the refrigerator or freezer for a minimum of 5 minutes

4.1.2 The temperature is recorded, dated and initialed by the operator on the Temperature Monitoring Sheet attached to the refrigerator (Figure 1).

4.2 Ovens

4.2.1 Prior to use, the oven is heated to the preset temperature

4.2.2 A thermometer is placed in the oven a minimum of 5 minutes to confirm oven temperature. The temperature, date, and time are noted in the operator's logbook. The thermometer is left in the oven

4.2.3 The sample is placed in the oven for a designated period of time.

4.2.4 Prior to sample removal, the thermometer is checked. The temperature, date, and time are noted in the operator's logbook.

4.3 Refrigerator, Freezer, and Oven Maintenance

Refrigerators, freezers, and ovens owned by the ESB are maintained and repaired by qualified repairmen in the NAVSURFWARCENDIV Crane Public Works Department. In the rare case when the Public Works Department cannot repair a refrigerator, freezer, or oven a contractor qualified by the malfunctioning device's manufacturer is brought in for the repair.

Figure 1

Refrigerator/Freezer Temperature Monitoring Sheet

Date	Temperature (°C)	Initials	Comments

Waste Segregation and Disposal

Explosive Sciences Branch

Naval Surface Warfare Center Crane

300 Highway 361

Crane, IN 47522

March 29, 2000

Approvals:

Name: Nancey Maegerlein
Title: Explosive Sciences Branch Manager
Signature: _____
Date: _____

Name: Jacquelyn Karty
Title: Chemist
Signature: _____
Date: _____

DRAFT

1.0 PURPOSE: This document outlines the procedures followed in the Explosive Sciences Branch (ESB) for waste segregation and disposal.

2.0 SCOPE: This document references NAVSURFWARCENDIV Instruction 5090.2B "Hazardous Waste Management Plan" for Crane Division, Naval Surface Warfare Center (NAVSURFWARCENDIV Crane) and briefly describes internal ESB procedures for waste segregation and disposal of common solvents.

3.0 NAVSURFWARCENDIV Instruction 5090.2B

NAVSURFWARCENDIV Instruction 5090.2B states NAVSURFWARCENDIV Crane's policy for the management and minimization of hazardous wastes in accordance with Federal and State regulations. All hazardous waste generated by the ESB are covered by this instruction.

3.1. Internal ESB Procedures for Waste Segregation and Disposal

The ESB follows all directives outlined in NAVSURFWARCENDIV Instruction 5090.2B. A general overview of hazardous waste segregation and disposal is provided here:

3.1.1. Hazardous waste is collected in glass bottles that have airtight lids. If the hazardous waste is incompatible with glass, plastic or metal containers (which ever is appropriate) may be used as long as the container has an airtight lid.

3.1.2. A sticker is placed on the bottle with the words "HAZARDOUS WASTE" clearly visible and the contents of the container are clearly written on the label.

3.1.3. When the container is full, the label is initialed and dated by the operator.

3.1.4. The operator logs the container in the Hazardous Waste Generation Log Book located in B2707. The container contents, start fill date, waste profile number (if applicable), and final fill date are entered.

3.1.5. The container is then removed from B2707 to the hazardous waste storage locker located outside of B2707.

3.1.6. The container is placed in secondary containment inside the hazardous waste storage locker.

3.1.7. Hazardous waste is removed by contractor personnel from the hazardous waste storage locker in accordance with NAVSURFWARCENDIV Instruction 5090.2B.

Controlling, Maintaining, and Monitoring Instrument Records, Instrument Logbooks, And Laboratory Notebooks

Explosive Sciences Branch

Naval Surface Warfare Center Crane

300 Highway 361

Crane, IN 47522

March 29, 2000

Approvals:

Name: Nancey Maegerlein
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Signature: _____
Date: _____

Name: Jacquelyn Karty
Title: Chemist
Signature: _____
Date: _____

DRAFT

1.0 PURPOSE: This document outlines the procedures followed in the Explosive Sciences Branch (ESB) for controlling, maintaining, and monitoring instrument records, instrument logbooks, and laboratory.

2.0 SCOPE: This document is specific to ESB analytical chemistry instrumentation (i.e. high performance liquid chromatographs (HPLC), gas chromatographs (GC), and GC/mass spectrometers (MS) systems) with regards to instrument records and logbooks. This document is applicable to all laboratory notebooks issued by the ESB.

3.0 CONTROLLING, MAINTAINING, AND MONITORING INSTRUMENT RECORDS

3.1 Controlling Instrument Records

3.1.1 Each instrument will be issued a bound notebook (a.k.a. instrument maintenance logbook) for the purpose of documenting instrument maintenance and performance.

3.1.2 Each instrument maintenance logbook will have the instruments manufacturer and model number written on the front cover.

3.1.3 On the first page, the following information will be listed:

3.1.3.1 The instrument's serial number and/or service contract number.

3.1.3.2 The instrument's software serial number and/or service contract number.

3.1.3.3 Any instrument accessory's serial number and/or service contract number.

3.1.3.4 The manufacturer's name, address, and technical support telephone number.

3.1.3.5 The service engineer's name and telephone number (if applicable).

3.1.4 On the second page, the maintenance schedule and location of the instrument's operation and repair manual(s) are listed.

3.2 Maintaining Instrument Records

3.2.1 It is the responsibility of the primary and alternate operators of the instrument to ensure detailed records of instrument maintenance and performance are maintained in the instrument maintenance logbook.

3.2.2 It is the responsibility of all users of the instrument to record any maintenance or repairs done by them and record information on the instrument's performance (i.e. results of standards run with corresponding file name) when they use it.

3.2.3 It is the responsibility of the primary and alternate instrument operators to detail any maintenance or repair performed by the instrument's service engineer. The primary and alternate instrument operators will file any loose-leaf records left by the service engineer under the instrument's name in a common filing cabinet accessible to anyone in the branch.

3.1 Monitoring Instrument Records

3.3.1 Instrument maintenance logbooks will be kept in the laboratory bench drawer (or nearest other enclosed location) closest to the instrument's location. This location will be clearly labeled.

3.3.2 It is the responsibility of the primary and alternate instrument operators to periodically check that the instrument maintenance logbook remains in this location and is up to date.

4.0 CONTROLLING, MAINTAINING, AND MONITORING INSTRUMENT LOGBOOKS

4.1 Controlling Instrument Logbooks

Each instrument will be issued an instrument logbook (a binder filled with appropriate pages to record sample information - see Figure 1)In addition to maintaining a list of all samples run on the instrument and the instrumental conditions, copies of all standards (and tune reports for GC/MS systems) will be kept in the back of the binder for instrument maintenance/performance information.

4.2 Maintaining Instrument Logbooks

4.2.1 It is the responsibility of the primary and alternate operators of the instrument to ensure records of all samples run on the instrument are maintained.

4.2.2 It is the responsibility of every instrument user to record the information in the instrument logbook listed in Section 4.2.3 and 4.2.4.

4.2.3 Instrument users will list the following information (minimum) in the Instrument Logbooks:

- 4.2.3.1 Date of Run
- 4.2.3.2 Samples/standards run
- 4.2.3.3 Corresponding File Names
- 4.2.3.4 Instrument conditions
- 4.2.3.5 Comments

4.2.4 Instruments users will print off the results of all standards (and tunes for GC/MS systems) run and place them in the back of the instrument logbook.

4.3 Monitoring Instrument Logbooks

4.3.1 Instrument logbooks will be kept in the same laboratory drawer as the Instrument maintenance logbooks.

4.3.2 It is the responsibility of the primary and alternate instrument operators to periodically check that the instrument logbook remains in this location and is up to date.

4.3.3 At no time may pages be removed from the instrument logbook except by the primary and alternate operators for data archiving.

4.3.4 Instrument logbook pages will be archived under the instrument's name in a common filing cabinet accessible to anyone in the branch. In addition, location of the archived computer data will be listed on the sheets. This data will remain on file in the ESB for 5 years.

5.0 CONTROLLING, MAINTAINING, AND MONITORING LABORATORY NOTEBOOKS

5.1 Controlling Laboratory Notebooks

5.1.1 Each analyst in the ESB will be issued a notebook with a serial number by the Quality Assurance/Quality Control (QA/QC) Officer.

5.1.2 A data base on the Code 405 file server will track which notebook has been issued to which analyst. This data base will be maintained by the QA/QC Officer.

5.1.3 New notebooks will not be issued until previous notebooks are full (see exemption in Section 5.1.4).

5.1.4 In rare cases when all the analysts on a major project agree, a notebook will be issued for the sole use of that project. The notebook will be the responsibility of the primary and alternate project leaders.

5.2 Maintaining Laboratory Notebooks

Each analyst is responsible for maintaining his or her notebook in a neat, orderly, and understandable fashion. Any errors made while entering data into the laboratory notebook will be crossed out with a single line, date, and initialed.

5.3 Monitoring Laboratory Notebooks

5.3.1 Laboratory notebooks will be turned in the QA/QC Officer when it is full.

5.3.2 The QA/QC Officer will file the notebook in a centralized location where it can be easily accessed if necessary.

5.3.3 The ESB Branch Manager, at her discretion, may audit laboratory notebooks at any time to ensure they are maintained in a neat, orderly, and understandable fashion.

Figure 1

Waters 2690 HPLC Instrument Logbook

Date	User	File Name	Sample	Instrument Conditions	Comments

Quality Assurance

Explosive Sciences Branch

Naval Surface Warfare Center Crane

300 Highway 361

Crane, IN 47522

April 10, 2000

Approvals:

Name: Nancey Maegerlein
Title: Explosive Sciences Branch Manager
Signature: _____
Date: _____

Name: Sara Poehlein
Title: Chemist
Signature: _____
Date: _____

DRAFT ONLY

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1.0 PURPOSE: This document outlines the procedures that will provide sound analytical chemical measurements. This plan is intended to provide the necessary guidance for the laboratory in the development of scientifically sound correct data of known precision and accuracy.

2.0 REPONSIBILITES AND GUIDELINES FOR PERSONNEL

The direct and ultimate responsibility for assuring data quality at the ESB laboratory rests with the ESB Laboratory Supervisor. The chemist/laboratory analyst will provide the first level of data review in the laboratory, however the ESB Laboratory Supervisor provides the final quality assurance/control of all data gathered or reported for compliance testing.

All personnel will strive to meet the requirements of the QA programs. All personnel will have access to procedure and technical documents and are encouraged to discuss the contents with the management at any time. Any changes suggested from these discussions must be approved by the laboratory QA officer and Laboratory Supervisor.

The QA officer will supervise the training and evaluation of all new personnel to ensure that everyone performing the analysis is thoroughly familiar with the program described in this manual. Documentation of training procedures for each individual will be kept in the personnel's file.

3.0 ORGANIZATION AND RESPONSIBILITY

3.1 PURPOSE

This section describes the responsibilities of each personnel. Their duties are outlined in this section and are graphically represented in Figure 3-1.

3.2 ORGANIZATIONAL CHART

3.2.1 Explosive Sciences Branch Laboratory Supervisor

Primarily responsible for ensuring the QA/QC program within the laboratory is in compliance and data quality adheres to this compliance. In delegating QA responsibilities/authorities, the ESB Laboratory Supervisor depends on the Quality Assurance Officer to coordinate all necessary QA requirements.

3.2.2 Quality Assurance Officer

This individual is responsible for the following:

1. Daily monitoring of quality control procedures.
2. Maintenance of appropriate QC charts and documents.
3. Document QC errors and work with the Laboratory Supervisor to ensure corrective action.
4. Initiate an in-house proficiency evaluation program involving blind samples.
5. Keep the Laboratory Supervisor apprised of the general state of the quality assurance program.
6. Review new technology, methods, and equipment and advises management as to such use, with respect to quality aspects.
7. Advises the purchasing agents with regard to the quality of purchased equipment, materials, reagents, and chemicals.

3.2.3 Assistant Quality Assurance Officer

Will help the QAO in implementing the quality assurance program and take over the duties of the QAO, as needed.

3.2.4 Senior Laboratory Analyst

Directs and oversees all laboratory personnel in the enactment of the required analytical procedures and necessary quality control activities. Assists the

QAO in fulfilling the objectives of the quality assurance plan.

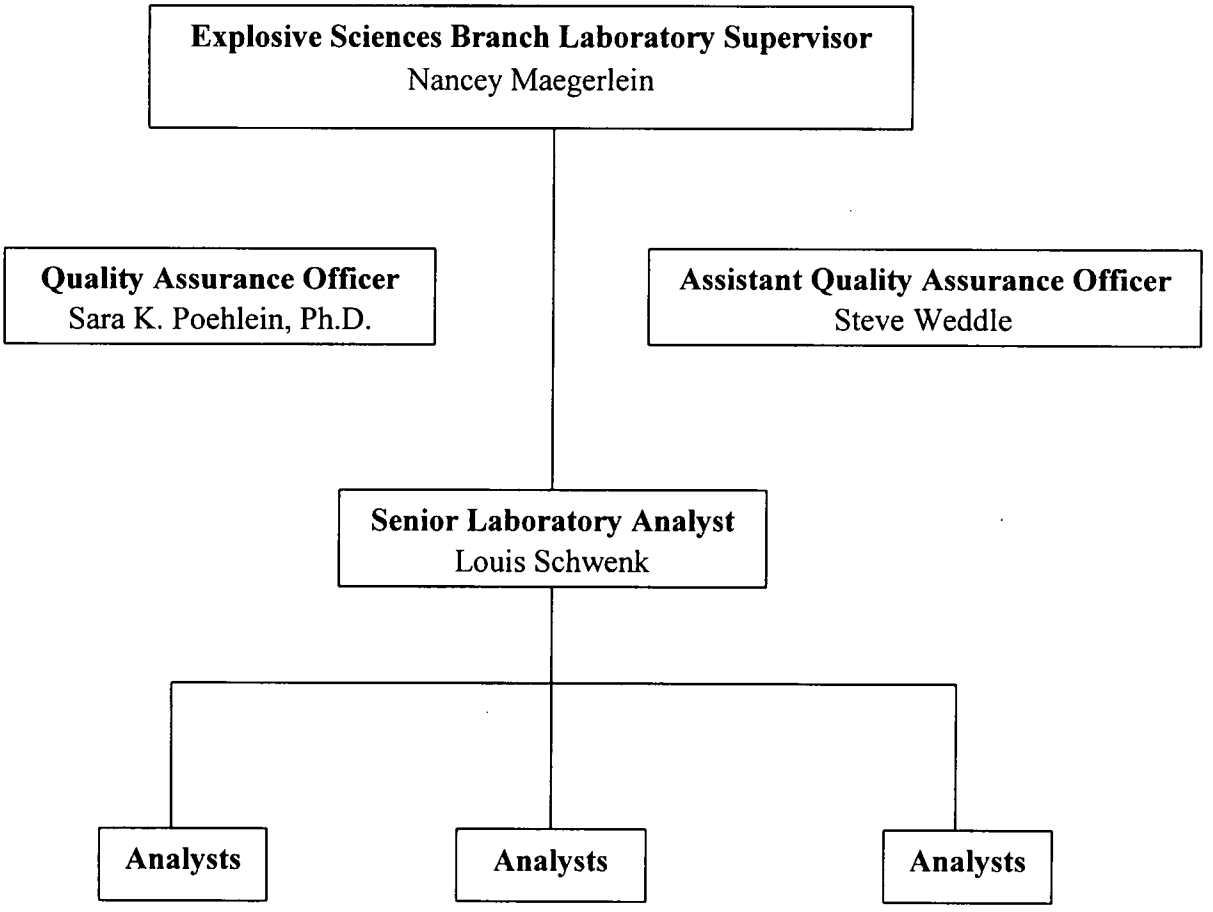
3.2.5 Laboratory Analyst

The laboratory analysts are responsible for the following:

1. Maintain proper log-in and sample tracking and active chain-of-custody files.
2. Verify the use of proper sample containers and preservatives of the incoming samples and provide appropriate written comments to document any discrepancies.
3. Provide for appropriate disposal of all samples after completion of the analysis.
4. Perform all analytical procedures according to approved protocol.
5. Make appropriate recommendations for correction and improvement, as may be necessary.

Figure 3-1

LABORATORY ORGANIZATION CHART



4.0 QUALITY ASSURANCE OBJECTIVE

The QA Plan is used to describe the quality assurance system followed by the ESB laboratory personnel. The policy of the ESB laboratory is to apply the quality system requirements to all sample analysis. The QA Plan provides personnel and customers of ESB with a description of the laboratory policy for maintaining an effective quality assurance program. The QA Plan is used as a guide to the continued maintenance and improvement of the quality service of the ESB laboratory.

5.0 QC CORRECTIVE ACTION

This section describes the procedure taken when there is a suspicion of nonconforming results, occurring during testing or analytical operations, data validation, or equipment failure. A corrective action program enables the analyst to handle performance failures at any point in data generation. This procedure is an indispensable tool for quality assurance and quality control management. A laboratory analyst or data user can use a corrective action report form to notify the laboratory Quality Assurance officer (or Laboratory Supervisor) of an issue (Figure 5-1).

Corrective action may be initiated by external sources. Unacceptable performance evaluations by regulatory agencies, poor correlation on field split samples, external audits, or questions concerning data quality by clients will lead to investigation and corrective action.

All corrective action results will be documented. Corrective action may be as simple as calibration, re-calibration, re-standardization, preparation of new standards, re-calculation of data, or repeating the analysis. Supervisor approval is required for corrective action involving the purchase of new standards, determining or accepting data involving interferences, and making requests for manufacturers service. The Corrective Action Form, shown in Figure 5-1, will serve as the means for communicating all corrective action to supervisors and QA officer.

Figure 5-1 Corrective Action Form

CORRECTIVE ACTION REPORT FORM NO._____	
TO: (ORGANIZATION) RECEIVED BY: Date:	
PREPARED BY: REVIEWED BY: Date:	
ACTIVITY:	LOCATION:
Where did out-of-control incident occur (laboratory name, address, telephone number, code)?	
When - did the incident occur? - was it corrected?	
What - was the name of the analysis? - was the problem with the test/control/instrument? - was the nature of the correction action? - will be done to prevent the reoccurrence of the problem?	
Why - did the incident happen (if a scientific explanation is available)?	
RESPONSE-SIGNATURE:	TITLE,CODE:
APPROVAL SIGNATURE:	TITLE,CODE:

6.0 BLIND SPIKE PROGRAM

The blind spike program will serve as the laboratory's internal audit, when performance evaluation or quality evaluation samples are unavailable for a specific project.

6.1 Requisition of Spike

All spikes will be prepared by the QA officer or the QA assistant. The analyst must provide the following information on the Blind Spike Form (Figure 6-1):

1. Name of analyst
2. Name of analysis
3. Solvent matrix
4. Concentration range of the spike
5. Date needed
6. Location of standards, necessary for preparation of spike

The Blind Spike Form must be submitted one week prior to the date needed, in order to ensure ample time to prepare the spike.

6.2. Blind Spike Documentation

6.2.1. All blind spike requests will be placed in a binder for documentation purposes.

6.2.2. The spikes will be prepared and recorded in a blind spike notebook. The notebook will record the exact procedure in which the spike is prepared.

6.2.3. After the analysis of the spike, the analyst must request the actual result of the spike from the QA officer or assistant.

Figure 6-1 Blind Spike Form

BLIND SPIKE FORM	
Analyst:	Date:
Analysis:	Date Needed:
Concentration Range:	
Matrix:	
Location of Standards:	

7.0 SOLVENT QC MONITORING FOR TRACE RESIDUE ANALYSIS

7.1 Lot testing of solvents

7.1.1 For each lot of solvents ordered, a single trace residue analysis will be performed.

7.1.2 The analyst shall aliquot approximately 2ml of solvent into a HPLC vial.

HPLC Parameters:
Injection = 30 microliters
Mobile Phase = 50% Methanol/ 50% water
Run Time = 6 minutes

7.1.3 Once the solvent trace residue analysis has been completed, the analyst must report the results in the TRACE RESIDUE ANALYSIS notebook.

8.0 PREPARATION, STORAGE, SHELF LIFE AND TRACEABILITY DOCUMENTATION OF STANDARDS AND REFERENCE MATERIALS

NAVSURWARCENDIVCRANE INSTRUCTION 4855.4 provides the details for the management of these materials.

9.0 OVERVIEW OF REVIEW AND APPROVAL PRACTICES FOR VALIDATABLE DATA PACKAGES

9.1 Organization and Responsibility

9.1.1 Verify that the Laboratory Supervisor is responsible for:

1. Design and implementation of the QA program
2. Designs and maintains a lab organization chart
3. Assists with review of final reports

9.1.2 Verify that the Quality Assurance officer is responsible for:

1. Daily monitoring of quality control procedures
2. Initiating in-house proficiency evaluation program involving blind samples
3. Keeping Laboratory Supervisor apprised of the general state of the QA program

4. Maintain QC records
5. Review final reports

9.1.3 Verify that the Senior Laboratory Analyst is responsible for:

1. Technical improvements
2. Monitor all technical systems for proper analytical operation
3. Print and Review final reports
4. Maintaining chain of custody documents

9.1.4 Verify that the analysts are responsible for:

1. Performance of analytical procedures according to protocol
2. Reporting of any out of control situations to the area supervisor
3. Evaluation of instruments and software
4. Maintenance of instruments
5. Reporting any problems that affect the quality of data immediately to the Senior Laboratory Analyst

9.2 Sample Custody

9.2.1 Verify that samples received by laboratory have a completed chain of custody with them.

9.2.2 Verify that samples are stored for at least three months before disposal.

9.2.3 Verify that worksheets are complete.

9.2.4 Verify that all data associated with a particular project is filed in a folder, secured, and accessed by approved personnel only.

9.3 Data Reduction, Validation, and Reporting

9.3.1 Verify that the analyst computes final results and record all related data on the appropriate worksheets.

9.3.2 Verify that the final report is reviewed by the Senior Laboratory Analyst, QA officer, and Laboratory Supervisor.

9.3.3 Verify that all data relating to an analytical project is stored for specified number of years, depending on the project.

Percent Moisture Analysis in Soil

Explosive Sciences Branch

Naval Surface Warfare Center Crane

300 Highway 361

Crane, IN 47522

April 1, 2000

Approvals:

Name: Nancey Maegerlein_
Title: Explosive Sciences Branch Manager
Signature:_____
Date: _____

Name: Jacquelyn Karty_
Title: Chemist_
Signature:_____
Date: _____

DRAFT

1.0 PURPOSE: This document outlines the procedures followed in the Explosive Sciences Branch (Code 4052) at Crane Division, Naval Surface Warfare Center (NAVSURFWARCENDIV Crane) for analyzing of the percent moisture in soil.

2.0 SCOPE: This document references the United States Environmental Protection Agency (USEPA) Method 3550: Sonication Extraction.

3.0 USEPA Method 3550

USEPA Method 3550 describes the procedures and conditions for determining the percent moisture in soil samples for sonication extraction. The Explosive Sciences Branch follows this procedure for determining moisture in soil.

3.1 Immediately after weighing the sample for extraction, 5 to 10 grams of the sample is weighed into a tared crucible. The weights of the sample and crucible are recorded in the analyst's laboratory notebook.

3.2 The sample is dried overnight at 105°C. The sample is allowed to cool in a desiccator before final weighing.

3.2 Percent moisture is calculated by the following equation:

$$\frac{\text{Gram (Sample)} - \text{Gram (Dry Sample)}}{\text{Gram (Sample)}} \times 100 = \%\ \text{Moisture}$$

3.3 All results are recorded in the analyst's laboratory notebook.

Determination of the Density of Soil Containing Organic Colorants by the Drive-Cylinder Method

CR4052-PD-012

Revision: 0

Explosive Sciences Branch
Navel Surface Warfare Center Crane
Code 4052
300 Highway 361
Crane IN 47522

Approvals:

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Title: Supervisory Chemist
Signature: _____
Date: _____

Name: Anndra Morgan
Title: Chemical Engineer
Signature: _____
Date: _____

DRAFT ONLY

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1.0 SCOPE AND APPLICABILITY

1.1 This test method will be used for the determination of in-place density of soil by the drive-cylinder method. The samples should be obtained from a relatively undisturbed area by driving a thin-walled cylinder into the soil followed by the subsequent activities for the determination of in-place density. Two procedures will be described in this document. Testing soil on the surface and testing soil below the surface.

1.2 This method is not appropriate and should not be used for sampling the following: organic soils, very hard soils that cannot be penetrated easily, soil that cannot be retained in the cylinders (low plasticity) or soil which contain appreciable amounts of course of granular material

1.3 This method is limited to obtaining samples for the determination of in-place density or water content of certain soils. This procedure is beyond the scope of the test method for obtaining undisturbed samples suitable for laboratory testing or determination on engineering properties.

2.0 HEALTH AND SAFETY WARNINGS

Standard environmental sampling and laboratory protective clothing and eye covering is required during the execution of this method.

3.0 PERSONNEL QUALIFICATIONS

This method is restricted to use by or under the supervision of analysts experienced in environmental sampling and laboratory practices. Each analyst must demonstrate the ability to generate acceptable results with this method.

4.0 APPARATUS AND MATERIALS

4.1 Density Tubes (minimum of six cylinders recommended)
- 4.1.1 4"(101.6mm) Diameter
- 4.1.2 0.033 cubic foot capacity

4.2 Hammer Assembly

4.3 Drive Head
- 4.3.1 4" (101.6 mm) diameter

4.4 Weight
- 4.4.1 23 lbs (10.4 kg)

4.5 Straightedge

4.5.1 Steel, approximately 1/8 by 1 ½ by 12 inch
4.5.2 One edge sharpened at about a 45° angle for trimming sample flush with cylinder.

4.6 Auger
4.6.1 Iwan or similar type for digging below shallow depths.

4.7 Shovel or spade
4.7.1 To be used for digging out the cylinders after they have been driven into the soil.

4.8 Balances
4.8.1 1-kg capacity with accuracy of 1.0-kg
4.8.2 500-g capacity with accuracy of 0.1-g
4.8.3 200-g capacity with accuracy of 0.01 g

4.9 Drying Oven
4.9.1 Thermostatically-Controlled
4.9.2 Able to maintain 110±5°C throughout chamber

4.10 Sample Containers
4.10.1 Water tight to contain samples and density tubes after sampling
4.10.2 Containers and lids for moisture content

4.11 Desiccater

4.12 Cooler
4.12.1 Kept between 3 to 30°C for storing testing can take place.

4.13 Miscellaneous Equipment
4.13.1 Brushes
4.13.2 Sledgehammer
4.13.3 Metal can with lids
4.13.4 Spoons
4.13.5 Calipers with an accuracy of 0.01 in (0.0025mm) for calibration

5.0 CALIBRATION

5.1 Before initial testing and after every 5th sample taken with the tube visually inspect the cutting edge of the density tubes for dulled or damaged edges

5.2 Before testing and after every 5th sample taken with the tube, determine the mass and volume of each density tube.

5.2.1 Mass Determination

5.2.1.1 Weigh and record the mass of the density tubes to the nearest 1g

5.2.2 Volume Determination

5.2.2.1 Measure and record the height of the density tubes at four equally spaced points to the nearest 0.01 in (0.254 mm).

5.2.2.2 Measure and record the diameter of the density tubes at the swaged-end at four equally spaced points to the nearest 0.01 in (0.254 mm).

5.2.2.3 Calculate and record the volume of each density tube to the nearest $0.01 cm^3$ ($0.16 mm^3$) using the following equation:

$$\text{Density tube volume} = \pi r^2 h$$

5.3 Permanently identify each density tube by a number that can be traced back to the calibration data.

6.0 SAMPLING

6.1 Sampling at or Near the Surface

6.1.1 For near-surface sampling (not more than 6 inches (1meter) in depth)

6.1.1.1 Brush all loose particles from surface.

Note:
Sample through a hole bored with an auger or dug by a shovel from which loosened material has been removed.

6.1.1.2 Obtain as level a surface as possible.

Note:
Surface may be leveled with the use of a bulldozer blade or other heavy equipment blades as long as the sample area and vicinity are not deformed, compressed, torn or otherwise disturbed.

6.1.1.3 Assemble drive head, hammer assembly and density tube with the sharpened edge on the surface to be sampled.

6.1.1.4 Drive the density tube into the ground by raising the hammer assembly and allowing it to fall until the top of the density tube is about ½ inch (13mm) below the surface.

6.1.1.4.1 Do not overdrive the density tube during sampling. Over driving may result in deforming of

compressing the sample and may provide erroneous results.

6.1.1.4.2 Discard the sample if overdriving is suspected or occurs and resample the soil.

Note:

Hold the drive rod in a steady and vertical position, keeping the drive head in contact with the density tube while driving the density tube into to ground.

6.1.1.5 Remove the drive head and carefully dig the density tube from the ground with a shovel.

6.1.1.5.1 Remove the soil from around the side of the density tube.

6.1.1.5.2 Undercut several inches below the density tube before lifting the tube from the ground.

6.1.2 For near-surface but below surface sampling (more than 6 inches (1meter) in depth)

6.1.2.1 Follow the same procedure listed is paragraph 8.1.1 removing move soil from around the density tube to properly undercut the density tube for removal from the ground.

6.1.3 After removal of density tube from soil, trim excess soil from around the side of the tube.

6.1.4 Use a straight to trim the end of the sample flush with the ends of the density tube.

6.1.5 If the tube is not full on the sample is not representative discard the sample and take another sample.

Note:

A satisfactory sample is composed of relatively undisturbed soil representative of the soil in place and shall not contain rocks, roots, or other foreign material.

6.1.6 If the density tube was deformed of damaged while driving is into to soil, discard the sample and repair of replace the tube and resample.

6.2 Sampling Below the Surface

6.2.1 Drill a hole using an auger to the elevation of the layer to be sampled.

6.2.2 Clean the bottom of the hole as well a possible with a cleaning auger or other suitable tool leaving the bottom of the hole fairly level.

6.2.3 Assemble the density tube and drive head, using extensions if necessary.

6.2.4 Lower density tube into the hole and place firmly on the layer to be sampled.

6.2.5 Drive the density tube into the soil by blows of a hammer on top of the drive rod until the top of the tube is about 1 inch (25mm) below the surface being sampled.

6.2.5.1 There is only about a 2 inches (50mm) clearance in the drive head. Be careful not to overdrive.

6.2.6 Remove the sample from the ground by rocking the drive rod back and forth until the sample is free.

6.2.7 Remove the assembly from the hole and carefully remove the cylinder from the drive head.

6.2.7.1 If the sample breaks from the ground slightly above the cutting edge, force the sample back into the density tube by carefully pressing the top of the sample against a flat surface.

6.2.8 Trim excess soil from the density tube using a straight edge until the sample if flush and plane with the ends of the tube.

6.2.9 If the density tube was deformed of damaged while driving is into to soil, discard the sample and repair of replace the tube and resample.

6.3 Immediately determine the mass and water content of the sample in according to paragraph 9.0 or place the density tube and sample in a sample container that will prevent soil and water loss no longer than 48 hours until the mass and water determination can be made.

6.3.1 The sample container shall be kept between 3 and 30°C and out of direct sunlight until it can be tested.

7.0 PROCEDURE

7.1 Mass Determination

7.1.1 Determine the mass of the density tube and the soil sample to the nearest 1 g and record.

7.2 Removal of the soil from the Density Tube.

7.2.1 Obtain a representative specimen for water content determination or use entire sample.

7.2.1.1 Samples should be as large a practical but in no case smaller than 10g.

7.3 Water Content

7.3.1 Weigh and record the mass of a clean and dry container and lid

7.3.2 Place the soil in container and place the lid on top of the container.

Note:
Larger containers should be used for large samples to air in sample drying

7.3.3 Determine and record the mass of the container, sample and lid.

7.3.4 Dry to a constant mass in drying oven maintained at 110±5°C.

7.3.4.1 Place the container the drying oven with the lid removed.

7.3.4.2 Allow to dry for 16 hours (overnight) in oven.

7.3.4.3 Remove container from drying oven and place in desiccator with lid in place for at least 1 hour.

7.3.4.4 Determine and record the mass using same balance that initial weights were taken on.

7.3.4.4.1 If not sure that sample is dry, place sample (uncovered) back in oven for no less that one half hour.

7.3.4.4.2 Remove and desiccate for 1 hour weigh and record. If weight indicates insignificant change (less than 0.1%) sample is dry.

8.0 CALULATIONS

8.1 Water Content

Calculate the water content using the following equation:

$$w = [W_1-W_2)/(W_2-W_c)] \times 100 = W_w/W_s \times 100$$

Where:
w = water content (%)
W_1 = mass of container and moist sample (g)
W_2 = mass of container and oven-dried sample (g)
W_c = mass of container (g)
W_w = mass of water (g) and
W_s = mass of solid particles (g)

8.2 Dry Mass

Calculate the dry mass with the following equation:

$$M_3 = [(M_1-M_2)/(100+w)] \times 100$$

Where:
M_3 = dry mass (g)
M_1 = mass of density tube and wet soil sample (g)
M_2 = mass of density tube (g) and
w = water content (%)

8.3 Dry Density

Calculate the dry density of the sample using the following equation:

$$\rho_d = (M_3/V)(3.810)$$

Where:
ρ_d = density in lb/ft^3
V = volume of density tube in in^3

9.0 REPORT

9.1 The Following shall be included in the report
- 9.1.1 Location.
- 9.1.2 Depth below ground surface or elevation of surface of both.
- 9.1.3 Dry density.
- 9.1.4 Water content.
- 9.1.5 Dimensions and volume of the density tube.
- 9.1.6 Visual description of the sample.
- 9.1.7 Comments on soil sample disturbance.

10.0 REFERENCES

DRAFT ONLY

10.1 ASTM Method D 2937, 1990, Standard Test Method for Density of Soil in Place by the Drive-Cylinder Method.

10.2 ASTM Method D 2216, 1990, Standard Test Method for Laboratory Determination of Water (Moisture) Contend of Soil, Rock, And Soil-Aggregate Mixtures.

Particle-Size Analysis of Soil Containing Organic Colorants

CR4052-PD-013

Revision: 0

Explosive Sciences Branch
Navel Surface Warfare Center Crane
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300 Highway 361
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1.0 SCOPE AND APPLICABILITY

This procedure covers the determination of particle size distribution of soil containing organic colorants. For distribution of particle sizes greater than 75µm (retained on the No. 200 sieve) will be determined by sieving. For distribution of particle sizes less than 75µm will be determined a sedimentation process with the use of a hydrometer.

2.0 HEALTH AND SAFETY WARNINGS

Standard laboratory protective clothing and eye covering is required during the execution of this method.

3.0 PERSONNEL QUALIFICATIONS

This method is restricted for use by or under the supervision of analysts experienced in laboratory practices. Each analyst must demonstrate the ability to generate acceptable results with this method.

4.0 APPARATUS AND MATERIALS

4.1 Balances
- 4.1.1 Sensitive to 0.01g
- 4.1.2 Sensitive to 1% of the mass of the sample to be weighed for weighing material retained on a No. 10 sieve.

4.2 Mortar and Rubber-Covered Pestle
- 4.2.1 Suitable for breaking up the aggregations of soil particles.

4.3 Sampler
- 4.3.1 Riffle sampler or sample splitter, for quartering the samples.

4.4 Mechanical Analysis Stirrer
- 4.4.1 Electric motor capable of turning vertical shaft at a speed no less than 10,000 rmp without a load.
- 4.4.2 Removable disc-shaped, nickel-plated steel mixing paddle

4.5 Hydrometer
- 4.5.1 ASTM
- 4.5.2 Conforming to hydrometer 151H or 152H in Specification E 100

4.6 Sedimentation Cylinder
- 4.6.1 Glass
- 4.6.2 18 inches (457 mm) high
- 4.6.3 21/2 inches (63.7 mm) in diameter
- 4.6.4 Graduation mark at 1,000 ml at 20 °C

4.7 Thermometer
4.7.1 Accurate to 1°F (0.5°C)

4.8 Sieves
4.8.1 Meet ASTM E11 requirements

4.9 Water Bath
4.9.1 Able to maintain a constant temperature at or near 68°F (20°C)

4.10 Beaker
4.10.1 250 ml Capacity

4.11 Volumetric Flask
4.11.1 1 L Capacity

4.12 Timing Device
4.12.1 Able to monitor time in seconds

4.13 Glass container for hygroscopic moisture

4.14 Drying Oven
4.14.1 Thermostatically-Controlled
4.14.2 Able to maintain 110±5°C throughout chamber

4.15 Rubber Stopper to fit top of sedimentation cylinder

4.16 Graduated Cylinder

5.0 REAGENTS

5.1 Sodium Hexametaphosphate (sodium metaphosphate)
5.2 Demineralized Water

6.0 DISPERSING AGENT

6.1 40 grams of sodium hexametaphosphate per liter of water.
6.1.1 Water used for test shall be brought to temperature the testing will be performed at (20°C) before use.

6.2 Label and Date Flask

NOTE:
Dispersing agent should be prepared at least once a month. The pH of the solution should be 8 or 9.

7.0 TEST SAMPLE

7.1 Sampling

7.1.1 Expose soil sample received from field to the air at room temperature until dried thoroughly.

7.1.2 Break up sample thoroughly in a mortar with a rubber-covered pestle.

7.1.3 Select a representative sample of the amount required to perform particle-size testing by the quartering method or use of a sampler.
7.1.3.1 115 grams of sandy soils passing through a No. 10 sieve
7.1.3.2 65 grams or silt of clay soils passing through a No. 10 sieve

7.2 Preparation of Test Sample

7.2.1 Weigh and record the soil obtained in paragraph 7.1.3. as the total test sample mass.

7.2.2 Separate the sample by sieving with a No. 10 (2.00 mm) sieve.

7.2.3 Grind the sample that remains on the No. 10 sieve in a mortar with a rubber-covered pestle until the aggregations of soil particles are broken up into the separate grains.

7.2.4 Separate the ground soil by sieving with a No. 10 sieve.

7.2.5 Wash the soil that remains after the second sieving free of all fine material.

7.2.6 Dry and Record weight after drying as the mass of course material.

7.2.7 Thoroughly mix together the soil that passed through the No. 10 sieve from both sieving steps (Para. 7.2.2 and 7.2.4)

7.2.8 Use the quartering method of a sampler to select about 115 grams of sandy soil or 65 grams of silt of clay soil.

7.2.9 Weigh and Record

8.0 PROCEDURE

8.1 Sieve Analysis of Sample retained on No. 10 sieve

8.1.1 Separate sample retained on the No. 10 sieve (from Para. 7.2.6) using the following sieves (or as many needed depending on sample):

3-in (75-mm)
2-in (50-mm)
1½-in (37.5–mm)
1-in (25-mm)
¾-in (19-mm)
3/8-in (9.5-mm)
No. 4 (4.75-mm)
No. 10 (2-mm)

8.1.1.1 Conduct sieving operation by using a lateral and vertical motion with the sieve, with the use of a jarring action to keep the sample moving continuously over the sieve surface.

NOTE:
Do not manipulate the sample in the sieve by hand.

8.1.1.2 Continue sieving until there is not more than 1 mass % of the residue on a sieve passes that sieve during 1 min of sieving.

8.1.2 Weigh and record the sample left of each sieve.

NOTE:
The sum of the sample retained on all the sieved should be close to the initial weight before sieving.

8.2 Hydrometer and Sieve Analysis of Sample Passing through the No. 10 Sieve

8.2.1 Determination of composite Correction For Hydrometer Reading

8.2.1.1 Equations for percentages of soil remaining is suspension are based on the use of distilled or demineralized water. The use of the dispersion agent in the water results in a liquid whose specific gravity is appreciable greater than that of distilled or demineralized water.

8.2.1.1.1 The soil hydrometers are calibrated at 68°F (20°C). Variations in temperature will results in inaccurate hydrometer readings. The accuracy increases as the temperature variation increases.

8.2.1.1.2 Hydrometers are graduated to be read at the bottom of the meniscus formed by the liquid on the

stem. Soil suspensions cannot be read at the bottom and have to be read at the top or the meniscus and a correction applied.

8.2.1.2 The net amount of the corrections for the three items enumerated is designated as the composite correction, and may be determined experimentally.

8.2.1.2.1 A graph or table of composite corrections for a series of 1° temperatures may be prepared and used a needed. Measurement of the composite corrections may be made at two temperatures spanning the range of expected test temperatures, and corrections for the intermediate temperatures calculated assuming a straight-line relationship between the two observed values.

8.2.1.3 Prepare 1000mL of liquid composed of distilled or demineralized water and dispersing agent in the same proportion as will prevail in the sedimentation (hydrometer) test. Place the liquid in a sedimentation cylinder and the cylinder in the constant-temperature water bath, set for one of the two temperatures to be used. When the temperature of the liquid becomes constant, insert the hydrometer, and, after a short interval to permit the hydrometer to come to the temperature of the liquid, read the hydrometer at the top of the meniscus Formed on the stem. For hydrometer 151H the composite correction is the difference between this reading and one: for hydrometer 152H it is the difference between the reading and zero. Bring the liquid and the hydrometer to the other temperature to be used, and secure the composite correction as before.

8.2.2 Hygroscopic Moisture

8.2.2.1 Weigh 10 to 15 g of the sample that passed through the No. 10 sieve (Para. 7.2.9) into a glass container.

8.2.2.2 Dry sample to a constant mass in a 230 ± 9°F (110 ± 5°C) drying oven.

8.2.2.3 Remove sample and place in decissicator for at least 1 hour.

8.2.2.4 Weigh and record weight

8.2.3 Dispersion of Soil Sample

8.2.3.1 Weigh Air-dried sample from Para. 7.2.9.

8.2.3.1.1 50 g for soil consisting of mostly clay and silt.

8.2.3.1.2 100 g for soil consisting of mostly sand.

8.2.3.2 Place sample in the 240-ml beaker and cover with 125-ml of the hexametaphosphate solution.

8.2.3.3 Stir until sample is thoroughly wetted, and allow to soak for 16 hour.

8.2.3.4 Stir sample with the mechanical stirrer for 1 min.

8.2.3.4.1 Transfer sample to dispersion cup washing residue from beaker with demineralized water.

8.2.3.4.2 Add demineralized water, if necessary, so that the cup is more than half full.

8.2.3.5 Proceed immediately with Para. 8.2.4

8.2.4 Hydrometer Test

8.2.4.1 Immediately after dispersion, transfer the soil-water slurry to the Sedimentation Cylinder.

8.2.4.2 Add demineralized water to the 1000-ml mark.

8.2.4.3 Place the rubber stopper in the end of the cylinder.

8.2.4.4 Turn the cylinder upside down and back for 1 minute.

8.2.4.4.1 Number of turns should equal to 60 counting the turning upside down and back as two turns.

8.2.4.4.2 Any soil remaining in the cylinder after the first few turns should be loosened by vigorous shaking of the cylinder while in the inverted position.

8.2.4.5 Take hydrometer reading at the following intervals, or as many as need setting the cylinder in the water bath between the 2-min and the 5-min reading.

Hydrometer Readings in minutes: 2, 3, 15, 30, 60, 250, and 1440

8.2.4.5.1 The hydrometer reading is taken by carefully inserting the hydrometer into the cylinder

20 to 25 seconds before to the approximate depth it will have when the reading is to be taken.

8.2.4.6 Immediately after reading is taken, carefully remove the hydrometer and place it with a spinning motion in a graduated cylinder of clean demineralized water

8.2.4.7 Take and record the temperature of the suspension after the hydrometer reading with the thermometer.

8.2.5 Sieve Analysis

8.2.5.1 After final hydrometer reading, transfer suspension to a No. 200 (75μm) sieve.

8.2.5.2 Wash the water until the wash water is clear.

8.2.5.3 Transfer material from sieve to a glass container of know weight.

8.2.5.4 Dry in an o en at 230 ± 9°F (119 ± 5°C) for 2 hours.

8.2.5.5 Perform sieve analysis on the dry portion using as many sieved as desired, or required for the material.

8.2.5.5.1 Conduct sieving operation by using a lateral and vertical motion with the sieve, with the use of a jarring action to keep the sample moving continuously over the sieve surface.

NOTE:
Do not manipulate the sample in the sieve by hand.

8.2.5.5.2 Continue sieving until there is not more than 1 mass % of the residue on a sieve passes that sieve during 1 min of sieving.

8.2.5.5.3 Weigh and record the sample left of each sieve.

9.0 CALULATIONS

9.1 Sieve Analysis Values for the Sample Courser than the No. 10 Sieve

9.1.1 Calculate the percentage passing the No. 10 sieve by dividing the mass passing the No. 10 sieve by the mass of soil originally split on the No. 10 sieve, and multiplying the result by 100. To obtain the mass passing the No. 10 sieve, subtract the mass retained on the No. 10 sieve from the original mass.

9.1.2 To secure the total mass of soil passing the No. 4 (4.75-mm) sieve, add to the mass of the material passing the No. 10 sieve the mass of the fraction passing the No. 4 sieve and retained on the No. 10 sieve.

9.1.3 To secure the total mass of the mass of the soil passing the 3/8-in sieve, add to the total mass of soil passing the No.4 sieve, the mass of the fraction passing the 2/8-in sieve and retained on the No. 4 sieve.

9.1.4 For the remaining sieves, continue the calculations in the same manner.

9.1.5 To determine the total percentage passing for each sieve, divide the total mass passing by the total mass of sample and multiple the results by 100.

9.2 Hygroscopic Moisture Correction Factor

9.2.1 The hydroscopic moisture correction factor is the ratio between the mass of the oven-dried sample and the air-dry mass before drying. It is a number less then one. Except when there is no hydroscopic moisture.

9.3 Percentages of Soil in Suspension

9.3.1 Calculate the oven-dry mass of soil in the hydrometer analysis by multiplying the air-dry mass by the hygroscopic moisture correction factor.

9.3.2 Calculate the mass of a total sample represented by the mass of soil used in the hydrometer test, by dividing the oven-dry mass used by the percentage passing the N0. 10 sieve, and multiplying the result by 100. This is the weight *W* in the equation for percentage remaining in suspension.

9.3.3 The percentage of soil remaining in suspension at the level at which the hydrometer measuring the density of the suspension may be calculated as follows:

9.3.3.1 For hydrometer 151H

$$P = [(100000/W) \times G/(G-G_1)](R-G_1)$$

9.3.3.2 For hydrometer 152H

$$P = (Ra/W) \times 100$$

Where:

a = Correction faction to be applied to the reading of hydrometer 152H. (Values shown on the scale are computed using a specific gravity of 2.65. Correction factors are given in Table 1),

P = Percentage of soil remaining in suspension at the level which the hydrometer measures the density of the suspension,

R = Hydrometer reading with composite correction applied,

W = Oven-dry mass of soil in a total test sample represented by mass of soil dispersed, g,

G = Specific gravity of the soil particles, and

G_1 = Specific gravity of the liquid in which soil particles are suspended. Use numerical valued of one in both instances in the equation. In the first instance any possible variation produces no significant effect, and in the second instance the composite correction for R in based on a value of one for G_1.

TABLE 1
Values of Correction Factor, α, for Different Specific Gravities of Soil Particles*

Specific Gravity	Correction Factor*
2.95	0.94
2.90	0.95
2.85	0.96
2.80	0.97
2.75	0.98
2.70	0.99
2.65	1.00
2.60	1.01
2.55	1.02
2.50	1.03
2.45	1.05

*For use in equation for percentage of soil remaining in suspension when using Hydrometer 152H.

9.4 Diameter of Soil Particles

9.4.1 The diameter of a particle corresponding to the percentage indicated be a given hydrometer reading shall be calculated using the following equation:

$$D = K(L/T)^{1/2}$$

Where:

D = Diameter of particle, mm

K = Constant depending on the temperature of the suspension and the specific gravity of the soil particles. Value of K for a range of temperatures and a specific gravities are given in Table 2

L = Distance from the surface of the suspension to the level at which the density of the suspension is being measured, cm. (For a given hydrometer and sedimentation cylinder, values vary according to the hydrometer readings, see Table 3.) and

T = Interval of time from beginning of sedimentation to the taking of the reading, min.

Table 2
Values of K for Use in Equation for Computing Diameter of Particle in Hydrometer Analysis

	Specific Gravity of Soil Particles								
°C	2.45	2.50	2.55	2.60	2.65	2.70	2.75	2.80	2.85
16	0.01510	0.01505	0.01481	0.01457	0.01435	0.01414	0.01394	0.01374	0.01356
17	0.01511	0.01486	0.01462	0.01439	0.01417	0.01396	0.01376	0.01356	0.01338
18	0.01492	0.01467	0.01443	0.01421	0.01399	0.01378	0.01359	0.01339	0.01321
19	0.01474	0.01449	0.01425	0.01403	0.01382	0.01361	0.01342	0.1323	0.01305
20	0.01456	0.01431	0.01408	0.01386	0.01365	0.01344	0.01325	0.01307	0.01289
21	0.01438	0.01414	0.01391	0.01369	0.01348	0.01328	0.01309	0.01291	0.01273
22	0.01421	0.01397	0.01374	0.01353	0.01332	0.01312	0.01294	0.01276	0.01258
23	0.01404	0.01381	0.01358	0.01337	0.01317	0.01297	0.01279	0.01261	0.01243
24	0.01388	0.01365	0.01342	0.01321	0.01301	0.01282	0.01264	0.01246	0.01229
25	0.01372	0.01349	0.01327	0.01306	0.01286	0.01267	0.01249	0.01232	0.01215
26	0.01357	0.01334	0.01312	0.01291	0.01272	0.01253	0.01235	0.01218	0.01201
27	0.01342	0.01319	0.01297	0.01277	0.01258	0.01239	0.01221	0.01204	0.01188
28	0.01327	0.01304	0.01283	0.01264	0.01244	0.01255	0.01208	0.01191	0.01175
29	0.01312	0.01290	0.01269	0.01249	0.01230	0.01212	0.01195	0.01178	0.01162
30	0.01298	0.01276	0.01256	0.01236	0.01217	0.01199	0.01182	0.01165	0.01149

Table 3
Values of Effective Depth Based on hydrometer and Sedimentation Cylinder of specified Sizes

Hydrometer 151H				Hydrometer 152H			
Actual Hydrometer Reading	Effective Depth, L, cm	Actual Hydrometer Reading	Effective Depth, L, cm	Actual Hydrometer Reading	Effective Depth, L, cm	Actual Hydrometer Reading	Effective Depth, L, cm
1.000	16.3	1.020	11.0	0	16.3	31	11.2
1.001	16.0	1.021	10.7	1	16.1	32	11.1
1.002	15.8	1.022	10.5	2	16.0	33	10.9
1.003	15.5	1.023	10.2	3	15.8	34	10.7
1.004	15.2	1.024	10.0	4	15.6	35	10.6
1.005	15.0	1.025	9.7	5	15.5	36	10.4
1.006	14.7	1.026	9.4	6	15.3	37	10.2
1.007	14.4	1.027	9.2	7	15.2	38	10.1
1.008	14.2	1.028	8.9	8	15.0	39	9.9
1.009	13.9	1.029	8.6	9	14.8	40	9.7
1.010	13.7	1.030	8.4	10	14.7	41	9.6
1.011	13.4	1.031	8.1	11	14.5	42	9.4
1.012	13.1	1.032	7.8	12	14.3	43	9.2
1.013	12.9	1.033	7.6	13	14.2	44	9.1
1.014	12.6	1.034	7.3	14	14.0	45	8.9
1.015	12.3	1.035	7.0	15	13.8	46	8.8
1.016	12.1	1.036	6.8	16	13.7	47	8.6
1.017	11.8	1.037	6.5	17	13.5	48	8.4
1.018	11.5	1.038	6.2	18	13.3	49	8.3
1.019	11.3			19	13.2	50	8.1
				20	13.0	51	7.9
				21	12.9	52	7.8
				22	12.7	53	7.6
				23	12.5	54	7.4
				24	12.4	55	7.3
				25	12.2	56	7.1
				26	12.0	57	7.0
				27	11.9	58	6.8
				28	11.7	59	6.6
				29	11.5	60	6.5
				30	11.4		

9.5 Sieve Analysis Values for Sample finer than No. 10 Sieve

9.5.1 Calculate the mass fraction that would have been retained on the No. 10 sieve had it not been removed.

9.5.1.1 This mass is equal to the total percentage retained on the No.10 sieve (100 minus total percentage passing) times the mass of the total sample represented by the mass of soil used and the result divided by 100.

9.5.2 Calculate the total mass passing the No. 200 sieve.

9.5.2.1 Ass together the fractional masses retained on all the sieved, including the No. 10 sieve, and subtract this sum from the mass of the total sample.

9.5.2.2 Calculate next the tot masses passing each of the other sieves in a manner similar to that given in Para. 12.

9.5.2.3 Calculate last the total percentages passing be dividing the total mass passing by the total mass of sample and multiply the result by 100.

10.0 REPORT

10.1 The following should be included in the report:

10.1.1 Maximum particle size,

10.1.2 Percentage passing (or retained on) each sieve, in tabular or graphical form,

10.1.3 Description of particles,

10.1.4 Specific gravity, if unusually high of low,

10.1.5 Any difficulty in dispersion the fraction passing the No. 10 sieve, and

10.1.6 The dispersion device used and the length of the dispersion period.

10.2

11.0 REFERENCES

11.1 ASTM Method D 422, 1990, Standard Method for Particle-Size Analysis of Soils

Determination of Organic Colorants in Environmental Matrices by High-Performance Liquid Chromatography

Explosive Sciences Branch
Naval Surface Warfare Center Crane
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Crane, IN 47522

May 9, 2001

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1.0 SCOPE AND APPLICABILITY

This method is intended for the analysis of organic colorants by high performance liquid chromatography (HPLC) using a Photodiode Array Detector (PDA). This method is used to determine the concentration of the following dyes in aqueous and solid matrices:

Aqueous Soluble			Organic Soluble		
Compound Name	Abbr.	CAS #	Compound Name	Abbr.	CAS #
Acid Blue 1	AB1	129-17-9	Disperse Blue 14	DB14	2475-44-7
Acid Blue 9	AB9	3844-45-9	Disperse Red 9	DR9	82-38-2
Acid Blue 45	AB45	2861-02-1	Disperse Violet 1	DV1	128-95-0
Acid Orange 10	AO10	1936-15-8	Solvent Green 3	SG3	128-80-3
Acid Red 64	AR64	NA	Solvent Orange 3	SO3	495-54-5
Acid Yellow 3	AY3	8004-92-0	Solvent Orange 7	SO7	3118-97-6
Acid Yellow 23	AY23	1934-21-0	Solvent Red 1	SR1	1229-55-6
Acid Yellow 73	AY73	518-47-8	Solvent Red 24	SR24	85-83-6
Basic Violet 10	BV10	81-88-9	Solvent Yellow 2	SY2	60-11-7
Basic Yellow 2	BY2	2465-27-2	Solvent Yellow 3	SY3	97-56-3
			Solvent Yellow 14	SY14	842-07-9
			Solvent Yellow 33	SY33	8003-22-3
			1-Aminoanthraquinone	1AQ	82-45-1
			2-Aminoanthraquinone	2AQ	117-79-3

1.1 This method provides a solid phase extraction procedure for dyes in surface or ground water.

1.2 These dyes may be dangerous. When handling samples and stock solutions, follow safety precautions listed in Section 4.0 and 5.0.

2.0 SUMMARY OF THE METHOD

2.1 This method provides HPLC conditions for the detection of ppb and low ppm levels of dyes in aqueous and solid matrices. Prior to using this method, appropriate sample preparation must be used.

2.2 This method utilizes a dual gradient, where flow and mobile phase composition are varied over time, to achieve sufficient separation of the target analytes.

2.3 This method provides two individual gradient methods for the separation of the analytes. The short gradient method (40 minutes total) provides for rapid separation of the analytes with the loss of resolution between two coeluting pairs of analytes (Acid Yellow

3/Acid Yellow 23 and Acid Blue 1/Disperse Violet 1). The long gradient method (70 minutes total) provides for the separation of the two coeluting pairs from the short gradient method. For soils, the long gradient method will only be run if one of the analytes in the coeluting pairs is detected in the short gradient method. The long gradient is used for aqueous samples because the percent recovery is biased high in the short gradient. Therefore, quantification of aqueous samples will be based on the long gradient.

2.4 Aqueous Sample Extraction: This method is an adaptation of a membrane solid phase extraction procedure for nonvolatile organics described in EPA Method SW-846-8330. Aqueous samples are extracted using a C18 extraction disk and tetrabutylammonium hydroxide. A small volume of methanol extracts the analytes from the C18 disk and is concentrated by rotary evaporation.

2.5 Soil and Sediment Sample Extraction: Solid samples are extracted with a 5:5:50 v/v mixture of tetrabutylammonium hydroxide (in methanol): water:methanol and filtered prior to analysis.

2.6 Acid Blue 45 exhibits significantly reduced recoveries in particulate-contaminated aqueous matrices. This is due to the preferential binding of Acid Blue 45 to particulates because of its high polarity.

2.7 Basic Yellow 2 results are biased high in both solid and aqueous matrices. While this method can confirm the presence of basic yellow 2 in a sample matrix, the quantitative results will be significantly higher than the actual amount in the matrix.

2.8 Acid Yellow 23 deviates significantly in ICV and CCV despite considerable effort to correct the problem. Therefore, Acid Yellow 23 is exempt from ICV and CCV criteria and corrective action.

3.0 DEFINITIONS

This section defines the terms and acronyms as used in this SOP.

PDA	Photodiode Array Detector
CCV	Continuing Calibration Verification - Standard that is injected at some prescribed frequency during the analytical run sequence to determine if the instrument is still calibrated.

ICV	Initial Calibration Verification – Standards that are injected at the beginning of a 24 analytical run sequence to determine if the instrument is still calibrated.
LC	Liquid Chromatography
RT	Retention Time - The time (in minutes) at which a target analyte elutes from the LC column.
RT Window	Retention Time Window - The +/- value which is applied to the ICV to establish the time range used to make tentative compound identifications.
UV/VIS	Ultraviolet/Visible absorption spectra

4.0 HEALTH AND SAFETY WARNINGS

4.1 Standard laboratory protective clothing and eye covering is required during the execution of this method.

5.0 CAUTIONS

5.1 Refer to the instrument manufacturer's manual for routine instrument precautions. This includes an awareness of moving parts and the potential for electrical shock.

5.2 Waste Disposal – All waste will be disposed of according to federal, state, and local regulations.

5.3 Routine and Preventative Maintenance

PREVENTIVE MAINTENANCE FOR ANALYTICAL INSTRUMENTS

Instrument	Preventive Maintenance	Maintenance Frequency
HPLC	Change filter frit in mixer.	As needed (when pressure builds)
	Change column pre-filter.	As needed (2-3 months)
	Rinse water pump with methanol, filter water, sonicate water intake filter frit.	Approximately weekly.
	Change pump seals.	As needed.

6.0 INTERFERENCES

6.1 Solvent, reagents, glassware and other sample processing hardware may yield discrete artifacts and/or elevated baselines, causing misinterpretation of the chromatograms. All of these materials must be demonstrated to be free from interferences.

6.2 Several dye compounds decompose rapidly when exposed to light. Standards must be kept in a dark cool area until used. Samples and sample extracts must be stored in a dark cool area until analyzed.

7.0 PERSONNEL QUALIFICATIONS

This method is restricted to use by or under the supervision of analysts experienced in the use of HPLC, skilled in the interpretation of chromatograms, and experienced in handling dye materials. Each analyst must demonstrate the ability to generate acceptable results with this method.

8.0 APPARATUS AND MATERIALS

8.1 HPLC System

- Waters 2690 Separations Module (auto-sampler, gradient pump)
- Waters 996 Photodiode Array Detector
- Waters Temperature Control Module
- 30 uL minimum volume sample loop
- Waters Millennium^{32} Chromatography Software

The system is equipped with a Waters 2690 Separation Module. The pump is capable of achieving 5000 psi. For the low concentration option, the detector must be capable of a stable baseline at 0.001 absorbance units full scale.

8.2 Other Equipment
- 8.2.1 Temperature controlled ultrasonic bath
- 8.2.2 Balance ± 0.0001 g
- 8.2.3 Oven

8.3 Materials
- 8.3.1 Disposable cartridge filters – 0.45 um teflon filter
- 8.3.2 Pipettes – Class A, glass, appropriate sizes
- 8.3.3 Pasteur pipettes
- 8.3.4 Vials – appropriate sizes with Teflon-lined cap
- 8.3.5 Disposable syringes – plastipak, 5 cc or equivalent
- 8.3.6 Volumetric flasks – appropriate sizes with ground glass stoppers, Class A.
- 8.3.7 Vacuum desiccator – glass
- 8.3.8 Mortar and pestle – ceramic
- 8.3.9 Sieve – 30 mesh

8.4 Preparation of Material:
- 8.4.1 Glassware will be washed in a phosphate-free detergent with tap water, followed by tap water rinse, and then a final deionized water rinse. Glassware used with organic solvents will be rinsed with a suitable organic solvent, followed by acetone, followed by tap water and then receive the above listed minimum wash. Prior to use all glassware will be rinsed three times with the solvent in use.

9.0 REAGENTS

9.1 Reagent grade inorganic chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.

- 9.1.1 Methanol (CH3OH), HPLC grade
- 9.1.2 Tetrabutylammonium Hydroxide (TBA) – HPLC grade, 1.0M in methanol
- 9.1.3 Organic-free reagent water. All references to water in this method refer to organic-free reagent water, as defined in SW846, Chapter One.

9.2 Stock Standard Solutions

9.2.1 Due to the rarity of some organic colorants, the standards may not be available from commercial sources, and therefore bulk lot samples can be used as standards,

9.2.2 Dry each solid analyte standard to constant weight in a vacuum desiccator in the dark. Place 0.0100 g (weighed to 0.0001 g) of a single analyte into an amber 100 mL volumetric flask and dilute to volume with methanol (for Solvent Red 24 weigh 0.0100 g in 250 mL). Invert flask several times and sonicate for twenty minutes until dissolved. Store in refrigerator at 4 °C in the dark. Calculate the concentration of the stock solutions from the actual weight used. Stock solutions may be used for up to one year.

9.3 Working Standards:

9.3.1 Calibration standards at a minimum of five concentration levels should be prepared for the initial calibration through dilution of the stock standard solutions. Calibration standards should contain multiple target analytes. It is recommended the analytes be grouped to aid in integration. Recommended groupings are as follows: Group 1 – AB45, AY23, AY73, AO10, BY2, DV1, SY33, and DB14; Group 2 – AR64, AB9, BV10, 2AQ, SO3, 1AQ, SY14, and SO7; Group 3 – SY3, BZ, AN, and SR1; Group 4 – DR9, SY2, SG3, and SR24. An example of the dyes concentration ranges can be found in Table 5. These solutions must be refrigerated and stored in the dark. Prepare the working calibration standards as follows:

Standard	Source Solution	Amount Added (ml)	Final Volume (mL)
Standard 5	A	10*	100
Standard 4	B	50	100
Standard 3	C	50	100
Standard 2	D	5	10
Standard 1	D	25	100

*add 20 mL for solvent red 24 and solvent green 3

Source Solution
A = Stock Standard (Each Dye)
B = Standard 5
C = Standard 4
D = Standard 3

9.3.2 For continuing calibration, run a midpoint standard of all dyes at the beginning of the day, and run one group of midpoint standards every 10 samples.

9.4 Surrogate Spiking Solutions

9.4.1 The analyst should monitor the performance of the extraction and analytical system as well as the effectiveness of the method in dealing with each sample matrix by spiking each sample, standard and reagent water blank with two surrogates (i.e., analyte not expected to be present in the sample). The surrogates selected for this method are benzanthrone and anthracene. Both are very strong UV absorbing analyte, yet are somewhat colorless.

9.5 Matrix Spiking Solutions

9.5.1 Prepare matrix-spiking solutions in methanol such that the concentration in the sample is ten times the MDL (See Tables 3 & 4). The matrix spiking solution will be made from the stock standard of the individual analytes chosen.

9.6 HPLC Mobile Phase

9.6.1 To prepare 1 L of 0.005M TBA mobile phase, dilute 5ml of 1.0M TBA in methanol to 1 L with methanol in a volumetric flask

10.0 SAMPLE COLLECTION, PRESERVATION, HANDLING AND RECEIPT

10.1 Upon sample receipt, the laboratory's sample custodian examines each cooler's custody seals to verify that they are intact and that the integrity of the environmental samples has been maintained. The sample custodian signs the COC report. The sample custodian then opens the cooler and measures its internal temperature. The temperature reading is noted on the accompanying COC report. The sample custodian then examines the contents of the cooler. Sample container breakages or discrepancies between the COC report and sample label documentation are recorded. All problems or discrepancies noted during this process are to be promptly related to the field team leader. Inter-laboratory COC procedures and specific procedures for sample handling, storage, disbursement for analysis, and remnant disposal will be followed as specified in the CR4052-PD-0500.

10.2 Samples are collected in amber glass containers with Teflon-lined caps. All samples and sample extracts are stored at 4° C $\pm$ 2° C. All samples and sample extracts must be stored in the dark until analysis to avoid decomposition of the dyes. Based on guidelines

listed in SW-846 for the extraction of non-volatile compounds, aqueous samples must be extracted within 7 days of collection and solid samples must be extracted within 14 days of collection. All extracts must be analyzed within 40 days of sample preparation.

11.0 PROCEDURE

11.1 Sample Preparation

11.1.1 Aqueous Samples: Solid-phase extraction

11.1.1.1 Using a 1 L volumetric flask, add 5 mL of tetrabutylammonium hydroxide (in methanol) by pipette.

11.1.1.2 Dilute to volume with aqueous sample. Mix the contents well before extraction.

11.1.1.3 If the sample contains sediment, pour the contents of the 1 L volumetric flask into a 1 L beaker and allow the sediment to settle to the bottom. Pour the water portion onto the solid phase extraction disk first, then add the sediment after the water has been pulled through but prior to extraction with methanol.

11.1.1.4 Pull sample through the C18 solid phase extraction disk using the vacuum manifold apparatus. A flow rate of 10 to 25 mL/min through the SPE disk is optimal for target analyte recovery. Record the flow rate through the extraction disk in the laboratory notebook.

11.1.1.5 Dry the disk using the vacuum manifold apparatus.

11.1.1.6 Switch the stopcock to the receiving vessel and pull through a minimum of 30 mL of methanol.

11.1.1.7 Evaporate to 1 mL using a rotary evaporator.

11.1.1.8 Transfer extract to a 10 mL volumetric flask. Rinse the round bottom flask with methanol and transfer rinse to the 10 mL volumetric flask. Dilute with methanol to volume.

11.1.2 Soil and Sediment Samples

11.1.2.1 Dry the soil samples in air at room temperature (or colder) to a constant weight, being careful not to expose the samples to direct sunlight. Grind and homogenize the dried sample thoroughly in an acetonitrile-rinsed mortar to pass a No. 18 sieve.

11.1.2.2 Place a 10.0-g sub-sample of each soil sample in a 100-mL volumetric flask. Add 1.0 mL of

surrogate spike solution. If the sample is a matrix spike/matrix spike duplicate, add 1.0 mL of the matrix spike solution. Add 5.0 mL of tetrabutylammonium hydroxide (in methanol) and 5.0 mL organic-free water. Place the stopper on the flask, shake for 1 minute, then place in a cooled ultrasonic bath for 0.5 hour.

11.1.2.3 After sonication, shake sample for 1 minute, then add 50.0 mL of methanol. Shake for 1 minute then place in a cooled ultrasonic bath for 1.0 hour.

11.1.2.4 Transfer approximately 4 mL of the slurry in a disposable syringe and filter it through a 0.45-micron nylon filter. Discard the first 1 mL and retain the remainder in a Teflon-capped vial for HPLC analysis.

11.2 Chromatographic Conditions: Short Gradient

11.2.1 Recommended Column: Waters Xterra MS, C18 column, 4.6mm x 100mm, 3.5um particle size

11.2.2 Mobile Phase: High Purity Water / 0.005 M Tetrabutylammonium Hydroxide (TBA) in Methanol

11.2.3 Dual Gradient: Run time = 35 min

11.2.3.1 Initial Parameters:
75:25 Water:TBA
1.0 mL/min
Column Temp = 30°C

11.2.3.2 Final Parameters:
0:100 Water:TBA
1.0 mL/min
Column Temp = 30°C

11.2.4 Mobile phase flow and composition are varied on a linear gradient throughout the analytical run. The instrument is allowed to come to equilibrium after returning to the initial parameters for 5 min prior to the next run.

11.2.5 Detector Conditions: PDA detector is set to scan from 211nm to 700nm. To maximize sensitivity, extract and integrate a separate chromatogram for each dye at the appropriate, most sensitive, analytical wavelength according to the following chart:

Water Soluble Dyes

Dye Name	Abbr.	Analytical Wavelength (nm)

Acid Blue 1	AB1	635
Acid Blue 9	AB9	628
Acid Blue 45	AB45	633
Acid Orange 10	AO10	485
Acid Red 64	AR64	524
Acid Yellow 3	AY3	383
Acid Yellow 23	AY23	404
Acid Yellow 73	AY73	493
Basic Violet 10	BV10	549
Basic Yellow 2	BY2	378

Organic Soluble Dyes

Dye Name	Abbr.	Analytical Wavelength (nm)
Anthracene	AN	251
Benzanthrone	BZ	399
Disperse Blue 14	DB14	638
Disperse Red 9	DR9	513
Disperse Violet 1	DV1	588
Solvent Green 3	SG3	635
Solvent Orange 3	SO3	408
Solvent Orange 7	SO7	499
Solvent Red 1	SR1	504
Solvent Red 24	SR24	519
Solvent Yellow 2	SY2	416
Solvent Yellow 3	SY3	385
Solvent Yellow 14	SY14	486
Solvent Yellow 33	SY33	381
1-Aminoanthraquinone	1AQ	481
2-Aminoanthraquinone	2AQ	299

Example UV/VIS spectra of each dye and its approximate retention time can be found in Attachment 1

11.3 Chromatographic Conditions: Long Gradient

11.3.1 Recommended Column: Waters Xterra MS, C18 column, 4.6mm x 100mm, 3.5um particle size

11.3.2 Mobile Phase: High Purity Water/0.005 M Tetrabutylammonium Hydroxide (TBA) in Methanol

11.3.3 Dual Gradient: Run time = 65 min

11.3.3.1 Initial Parameters:
75:25 Water:TBA
0.7 mL/min
Column Temp = 30°C

11.3.3.2 Final Parameters:
0:100 Water:TBA
1.0 mL/min

Column Temp = 30°C

11.3.4 Mobile phase flow and composition are varied on a linear gradient throughout the analytical run. The instrument is allowed to come to equilibrium after returning to the initial parameters for 5 min prior to the next run.

11.3.5 Detector Conditions: Same as those described in Section 11.2.5.

11.4 Calibration of HPLC:

The entire HLPC system is allowed to warm up for a minimum of 30 minutes. During this time the mobile phase, at the initial gradient conditions, should be pumped through the column.

11.4.1 Initial Calibration

11.4.1.1 Injections of each calibration standard over the concentration range of interest are preformed. Peak heights and peak areas are obtained for each analyte at the appropriate wavelength.

11.4.2 Daily Calibration

11.4.2.1 Analyze the midpoint calibration standards along with an instrument blank at the beginning of each day and after each set of 10 samples or less. Obtain the response factor for each analyte from the mean peak heights or peak areas and compare it with the response factor obtained for the initial calibration. The mean response factor for the daily calibration must agree within ±25% of the response factor of the initial calibration. The same criterion is required for subsequent standard responses. If this criterion is not met, a new initial calibration must be obtained. Note exemption in Section 2.8.

11.5 HPLC Analysis

11.5.1 Analytical Sequence

All acceptable samples must be analyzed within a valid analysis sequence as given below:

Initial Calibration:

Injection Number	Material Injected
1	Instrument Blank

2 – 21	Five Initial Calibration Standards
22	Instrument Blank at end of initial calibration

Daily Analysis Sequence:

Time	Injection Number	Material Injected
0hr	1	Instrument blank
	2 – 5	Midpoint standard of all dyes (ICV)
	6	Instrument Blank
	7	LCS
	8 – 18	Sample Analyses
~12 hr	19	Instrument Blank
	20	Mid-level CCV of one group of dyes
	21	LCS
	22 – 32	Sample Analyses
~12 hr	33	Instrument Blank
	34	Mid-level CCV of one group of dyes
	Etc.	

After the initial calibration, the analysis sequence may continue as long as acceptable instrument blanks and continuing calibration verifications are analyzed at the required frequency. This analysis sequence shows only the minimum required blanks and standards. More blanks and standards may be run at the discretion of the analysts; these must also satisfy the criteria presented in Section 12 in order to continue the run sequence.

An analysis sequence must include all required matrix spike/matrix spike duplicate and method blank analyses. The analysts may decide at what point in the sequence they are to be analyzed.

11.5.2 Laboratory Control Sample (LCS)

11.5.2.1 The LCS (reagent blank spike) should be prepared separately from the standards and include at least 6 of the dyes, 3 water soluble and 3 organic soluble. The concentration of the dyes should be approximately 500 µg/L.

11.5.3 Follow the guidelines found in SW846, Method 8000 for the following parameters.

11.5.3.1 Retention Windows

11.5.3.1.1 Retention time windows are established by performing 7 replicate injections over a 72

hour period. Retention time windows are set at 3 times the standard deviation of the replicate injections.

11.5.3.2 Any modifications to the RTW

11.5.3.2.1 If the retention time of any analyte in the standards at the beginning of the analytical shift does not fall within the ±3 standard deviation window, then a new initial calibration is necessary unless system maintenance corrects the problem.

11.5.3.3 Initial Calibration

11.5.3.3.1 The initial calibration involves the analysis of standards containing each one of the analytes at five concentrations over the working range of the instrument.

11.5.3.4 Initial Calibration Verification

11.5.3.4.1 The initial calibration is verified at the beginning of each 12-hour analytical shift. The process involves the analysis of the mid-point standard for each group of dyes. If the response for the analyte is within ±25% of the response obtained during the initial calibration, then the initial calibration is considered still valid. Note exemption in Section 2.8.

11.5.3.5 Instrument Blank

11.5.3.5.1 An instrument blank is analyzed prior to injection of the standards, after the analysis of all standards, and after every 10 samples.

11.5.3.6 Continuing Calibration Verification

11.5.3.6.1 The continuing calibration verification process involves the analysis of the mid-point standard for one group of dyes. If the response for the analyte is within ±25% of the response obtained during the initial calibration, then the initial calibration is considered still valid. The CCV is analyzed every 10 samples. Note exemption in Section 2.8.

11.5.3.7 Sample Analysis

11.5.3.7.1 An autosampler is used to inject 10 to 100 µL of a sample. Samples are analyzed according to the sequence in 11.5.1.

11.5.4 Compound Identification

11.5.4.1 Compound identification is achieved through spectral matching with known standards of the dyes.

11.5.4.2 In the event of co-elution problems, the analyst will attempt to separate the co-eluting compounds using either the short or the long gradient as appropriate. Re-analysis of the sample to address co-elution problems will occur only if a target dye is positively identified (by retention time and spectra) and is co-eluting with another positively identified target dye. All samples must be analyzed within a valid analysis sequence as defined in Section 11.5.

11.5.5 Compound Quantification

11.5.5.1 Data Acquisition, Calculations and Data Reduction

11.5.5.1.1 All data acquisition, chromatogram integration, calibration curve calculation and unknown concentration calculation is preformed utilizing the Waters Millennium [32] chromatography software.

11.5.5.1.2 Manual calculations such as standard deviation and percent recovery will be performed utilizing a spreadsheet.

11.5.6 Data Management and Records Management

11.5.6.1 All documents are managed according to CR4052-PD-0100 and CR4052-PD-0150. Raw data is stored on using the Millennium 32 chromatography software. The data is backed-up monthly.

12 QUALITY CONTROL AND QUALITY ASSURANCE

12.1 Initial Calibration

12.1.1 Criteria: Analyze standards at five concentrations over working range of instrument. Linearity is determined by an RSD $\leq$ 30% or a coefficient of determination greater or equal to 0.99

12.1.2 Corrective Action: If the initial calibration does not meet the criteria for linearity, reduce/increase the concentration of the upper/lower standards until the criteria is meet.

12.2 Initial Calibration Verification

12.2.1 Criteria: The initial calibration is verified at the beginning of each 12-hour analytical shift. A mid-point standard for each group of dyes is analyzed. If the response for the analyte is within ±25% of the response obtained during the initial calibration, then the initial calibration is considered still valid. Up to 5 dyes are allowed to be outside this tolerance for the initial calibration to be considered still valid. Note exemption in Section 2.8.

12.2.2 Corrective Action: If the initial calibration is no longer valid, a new initial calibration must be performed unless system maintenance corrects the problem.

12.3 Continuing Calibration Verification

12.3.1 Criteria: The continuing calibration verification process involves the analysis of a mid-point standard for one group of dyes. If the response for the analyte is within ±25% of the response obtained during the initial calibration, then the initial calibration is considered still valid. Up to 2 dyes are allowed to be outside this tolerance for the initial calibration to be considered still valid. The CCV is analyzed every 10 samples. Note exemption in Section 2.8.

12.3.2 Corrective Action: If the initial calibration is no longer valid, a new initial calibration must be performed unless system maintenance corrects the problem.

12.4 Instrument Blank

12.4.1 Criteria: An instrument blank is analyzed prior to injection of the standards, after the analysis of all standards, and after every 10 samples. The results of the instrument blank should be less then the IDL.

12.4.2 Corrective Action: If the instrument blank does not meet the acceptance criteria, then the source of the contamination will be located and reduced. The samples associated with the contaminated instrument blank will be re-analyzed.

12.5 Method Blank

12.5.1 Criteria: A clean sample will be extracted using the appropriate procedure and analyzed. The results of the method blank should be less then the MDL or less than 5% of the sample result for the same analyte. A method blank will be prepared for each group of 20 samples.

12.5.2 Corrective Action: If the method blank does not meet the acceptance criteria, then the source of the contamination will be located and reduced. The samples associated with the contaminated method blank will be re-extracted and re-analyzed.

12.6 Laboratory Control Sample

12.6.1 Criteria: A clean matrix similar to the sample matrix and of the same weight or volume will be spiked with the same analytes at the same concentrations as the matrix spike, extracted using the same method, and analyzed. When the results of the matrix spike analysis indicates a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

12.6.2 Corrective Action: If the LCS does not meet the acceptance criteria, the source of the problem will be located and corrected.

12.7 Matrix Spike

12.7.1 Criteria: A sample matrix will be spiked with a minimum of 2 water-soluble dyes, 2 organic-soluble dyes, and the surrogate standards, extracted using the same method, and analyzed. Percent recovery is calculated.

12.7.2 Corrective Action: If the matrix spike indicates there may be interference from the matrix, then the LCS will be used to verify that the laboratory can perform the analysis in a clean matrix.

12.8 Matrix Spike Duplicate

12.8.1 Criteria: The same matrix used for the matrix spike will be spiked with the same analytes at the same concentrations as the matrix spike. Percent recovery is calculated

12.8.2 Corrective Action: If the matrix spike indicates there may be interference from the matrix, then the LCS will be used to verify that the laboratory can perform the analysis in a clean matrix.

12.9 Surrogate Standards

12.9.1 Criteria: A solution of benzanthrone and anthracene will be spiked on the sample matrix. Percent recovery is calculated.

13 REPORTS

13.1 Electronic Deliverables: Final results will be provided in a Microsoft Excel format. The following information will be included in the Excel sheet: sample number, laboratory identification, laboratory name, associated blank, quality control type, sample date, receipt date, extraction date, analysis date, run number, parameter, CAS number, method, laboratory results, instrument detection limits,

method detection limits, percent moisture, percent recovery of surrogates, and comments. Another Excel sheet will report RSD and coefficients of determination for the initial calibration curves, and tabulated results of the daily and continuing calibration standards.

13.2 Full data package: In addition to the electronic deliverables, the full data package will include chromatograms, HPLC concentration results, and UV-Vis spectra for the initial calibration, daily calibration, continuing calibration, instrument blanks, method blanks, and each sample. Full package will be provided when there is a positive detection of a target analyte.

13.3 Abbreviated data package: In addition to the electronic delieverables, the field screening data package will include chromatograms and HPLC concentration results for the initial calibration, daily calibration, and continuing calibration. Chromatograms for the samples will be provided to prove the absence of detectable dyes. The abbreviated data package will be provided when no target analyte is detected in the sample.

13.4 If an analyte is noted on the chromatogram but can not be confirmed by its UV-Vis spectra, then "NSC" (no spectra confirmation) will be noted in the comments section of the Electronic Deliverables

14 REFERENCES

14.2 Wegener, J.W., J.C. Klamer, H. Govers, and U.A. Th. Brinkman (1987) Determination of Organic Colorants in Cosmetic Products by High Performance Liquid Chromatography.

14.3 Chromatographia, 24: 865-875.Garrison, A.W., G.L. Baughman, E.J. Weber, R.L. Adams, and M.S. Brewer (1992) Fate of Colored Smoke Dyes. Army Project Order 88PP8863.

14.4 Alcantara-Licudine, J.P., M.K. Kawate, and Q.X. Li (1997) Method for the Analysis of Phloxine B, Uranine, and Related Xanthene Dyes in Soil Using Supercritical Fluid Extraction and High Performance Liquid Chromatography. Journal of Agricultural Food Chemistry, 45: 766-773.

14.5 Gagliardi, L., G. Cavazzutti, A. Amato, A. Basili, and D. Tonelli (1987) Identification of Cosmetic Dyes by Ion Pair Reversed Phase High Performance Liquid C hromatography. Journal of Chromatography, 394: 345-352.

14.6 Eakes, W., J. Heath, M. Cavit, K. Rimm, W. Powers, C. Connelly, J. Anderson, M. Johnson (1983) Initial Assessment Study of Naval Weapons Support Center Crane, Indiana. NEESA 13-003.

14.7 U.S. Environmental Protection Agency, Method SW-846-8330.

14.8 U.S. Environmental Protection Agency, Method SW-846-8000.

14.9 U.S. Environmental Protection Agency, Method SW-846-3500.

14.10 U.S. Environmental Protection Agency, Method SW-846-3550.

15 ATTACHMENTS

CR4052-PD-2500(1) Stock Standards Data Sheet

CR4052-PD-2500(2) Working Standards Data Sheet

CR4052-PD-2500(3) Surrogate and Matrix Spike Data Sheet

CR4052-PD-2500(4) Aqueous Extraction Data Sheet

CR4052-PD-2500(5) Solid Extraction Data Sheet

16 REVISION BLOCK

REV	DATE	REVISION	AUTHOR
0	5/09/01	Original Issue	JJK

TABLE 1
Quality Control Limits
Matrix Spike/Matrix Spike Duplicate Samples and Surrogate Spikes
Dye Analyses by the Short Gradient
Naval Surface Warfare Center
Crane, Indiana

Chemical	Solid Matrix	
	Accuracy (%R)	Precision (RPD)
Acid Blue 9	3-9	35
Acid Blue 45	46-75	35
Acid Orange 10	191-213	35
Acid Red 64	61-69	35
Acid Yellow 23	55-74	35
Acid Yellow 73	61-68	35
1-Aminoanthraquinone	73-94	35
2-Aminoanthraquinone	42-46	35
Basic Violet 10	94-105	35
Disperse Blue 14	34-73	35
Disperse Red 9	40-48	35
Disperse Violet 1	45-68	35
Solvent Green 3	25-72	35
Solvent Orange 3	51-54	35
Solvent Orange 7	58-94	35
Solvent Red 1	25-88	35
Solvent Red 24	TBD	TBD
Solvent Yellow 2	41-47	35
Solvent Yellow 3	210-225	35
Solvent Yellow 14	41-44	35
Solvent Yellow 33	35-41	35
Anthracene	42-57	NA
Benzanthrone	99-113	NA

NA : Not applicable
QC limits are based on the seven low spike samples analyzed during method performance/validation. Matrix spike QC limits are the same as laboratory control sample QC limits. Limits are not presented for aqueous samples since they will be quantitated using the long gradient.

TABLE 2
Quality Control Limits
Matrix Spike/Matrix Spike Duplicate Samples and Surrogate Spikes
Dye Analyses by the Long Gradient
Naval Surface Warfare Center
Crane, Indiana

Chemical	Aqueous Matrix (1)	
	Accuracy (%R)	Precision (RPD) (2)
Acid Blue 9	16-113	37
Acid Blue 45	0-45	53
Acid Orange 10	50-114	20
Acid Red 64	43-109	22
Acid Yellow 23	48-111	20
Acid Yellow 73	47-110	20
1-Aminoanthraquinone	41-96	20
2-Aminoanthraquinone	36-87	21
Basic Violet 10	46-111	21
Disperse Blue 14	35-109	26
Disperse Red 9	46-115	22
Disperse Violet 1	46-100	20
Solvent Green 3	2-84	47
Solvent Orange 3	41-92	20
Solvent Orange 7	37-65	20
Solvent Red 1	38-74	20
Solvent Red 24	34-102	25
Solvent Yellow 2	47-104	20
Solvent Yellow 3	49-116	20
Solvent Yellow 14	42-88	20
Solvent Yellow 33	46-106	20
Anthracene	0-106	NA
Benzanthrone	32-120	NA

NA: Not Applicable
Solid samples will be quantitated by the short gradient.

1. QC limits based on 11 low spike DI water samples analyzed during Rounds 2 and 3 of the Method Validation Study. New limits for groundwater will be developed when sufficient data has been generated.

2. RPD limits of 20% for aqueous and 35% for solids were substituted for the calculated value in instances where the calculated RPD was less than the 20% or 35% limit.

TABLE 3
Analytical Methods and Limits of Detection For the Short Gradient Naval Surface Warfare Center Crane, Indiana

Chemical	Laboratory MDL		Laboratory RL		Risk-Based Target Level	
	Aqueous (ug/L)	Solid (ug/kg)	Aqueous (ug/L)	Solid (ug/Kg)	Aqueous (ug/L)	Solid (ug/kg)
Acid Blue 1	55.3	558.9	26.63	15978	450	10000
Acid Blue 9	27.5	141.8	22.63	13578	230000	1630000
Acid Blue 45	79.1	791.6	23.25	13950		
Acid Orange 10	39.3	522.7	25.13	15078	100	6460
Acid Red 64	17.1	240.7	62.5	37500		
Acid Yellow 3	104.3	149.0	22.88	13728		
Acid Yellow 23	92.7	403.7	25.13	15078		
Acid Yellow 73	19.7	179.9	22.63	13578		
1-Amino-anthraquinone	18.7	425.6	16.06	9636		
2-Amino-anthraquinone	10.9	138.7	23.36	14016	2	14700
Basic Violet 10	20.8	177.2	19.88	11928	570	12000
Basic Yellow 2	384.6	1350.6	23.13	13878	60	1300
Disperse Blue 14	60.7	912.5	18.69	11214		
Disperse Red 9	20.7	213.1	16.51	9906		
Disperse Violet 1	35.5	467.8	16.26	9756		
Solvent Green 3	45.2	1156.9	18.38	11028		
Solvent Orange 3	28.7	65.3	15.99	9594	600	129000
Solvent Orange 7	97.4	799.3	18.00	10800	730	52000
Solvent Red 1	84.6	2304.2	28.99	17394		
Solvent Red 24	27.9	0	7.1	4260		
Solvent Yellow 2	23.7	192.5	19.95	11970	0.01	110
Solvent Yellow 3	54.1	391.2	18.88	11328	0.02	130
Solvent Yellow 14	33.7	80.7	17.70	10620	460	3230
Solvent Yellow 33	20.5	136.5	17.48	10488		
Anthracene	115.9	331.9	25.96	15576		
Benzanthrone	30.4	176.2	15.2	9120		

The risk-based screening levels were developed by USEPA Region 5 specifically for this project

TABLE 4
Analytical Methods and Limits of Detection For the Long Gradient Naval Surface Warfare Center Crane, Indiana

Chemical	Laboratory MDL		Laboratory RL		Risk-Based Target Level	
	Aqueous (ug/L)	Solid (ug/kg)	Aqueous (ug/L)	Solid (ug/Kg)	Aqueous (ug/L)	Solid (ug/kg)
Acid Blue 1	43.8 (1)	523.4	26.63	15978	450	10000
Acid Blue 9	25.1	89.5	22.63	13578	230000	1630000
Acid Blue 45	26.7	407.5	23.25	13950		
Acid Orange 10	13.0	262.0	25.13	15078	100	6460
Acid Red 64	12.1	325.8	62.5	37500		
Acid Yellow 3	24.6 (1)	2029.5	22.88	13728		
Acid Yellow 23	12.8	512.8	25.13	15078		
Acid Yellow 73	13.5	169.0	22.63	13578		
1-Amino-anthraquinone	12.2	242.5	16.06	9636		
2-Amino-anthraquinone	12.6	230.0	23.36	14016	2	14700
Basic Violet 10	15.0	146.0	19.88	11928	570	12000
Basic Yellow 2	170	1334.1	23.13	13878	60	1300
Disperse Blue 14	16.7	390.9	18.69	11214		
Disperse Red 9	14.8	176.7	16.51	9906		
Disperse Violet 1	11.1	950.1	16.26	9756		
Solvent Green 3	18.5	697.9	18.38	11028		
Solvent Orange 3	11.7	56.7	15.99	9594	600	129000
Solvent Orange 7	6.1	176.2	18.00	10800	730	52000
Solvent Red 1	7.9	2295.6	28.99	17394		
Solvent Red 24	2.9		7.1	4260		
Solvent Yellow 2	11.8	221.0	19.95	11970	0.01	110
Solvent Yellow 3	16.2	194.6	18.88	11328	0.02	130
Solvent Yellow 14	13.5	94.7	17.70	10620	460	3230
Solvent Yellow 33	13.7	137.4	17.48	10488		
Anthracene	18.4	236.6	25.96	15576		
Benzanthrone	13.1	83.1	15.2	9120		

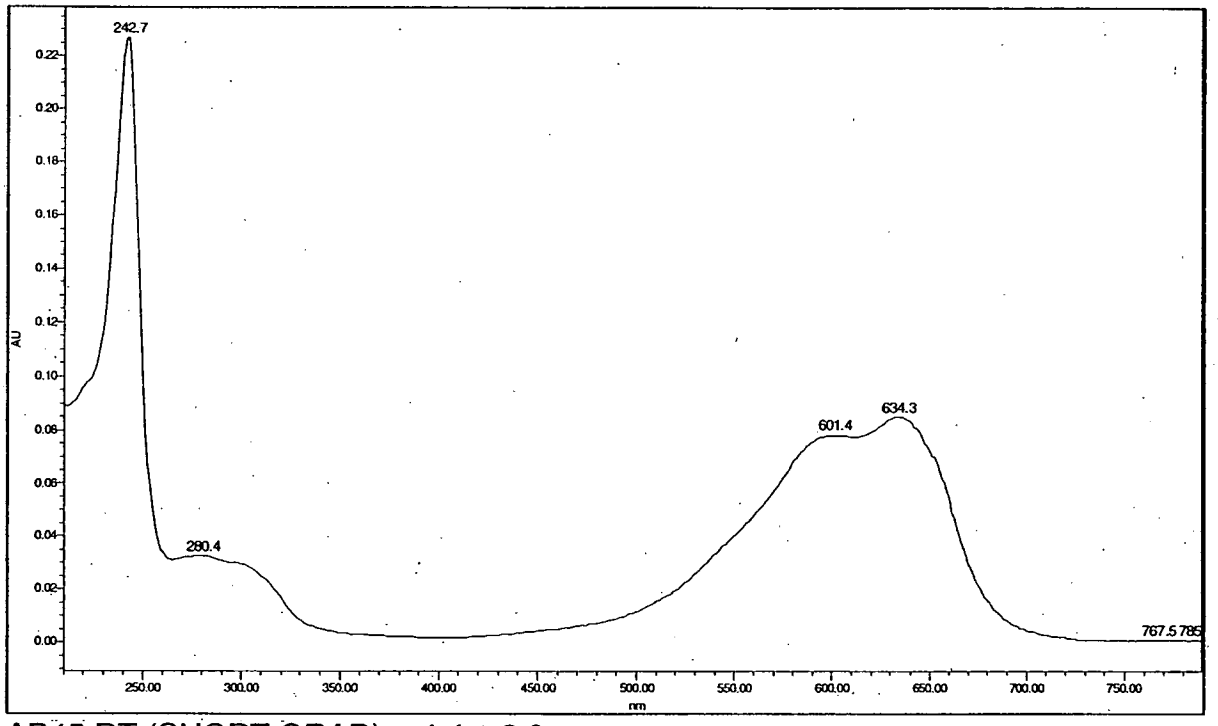
The risk-based screening levels were developed by USEPA Region 5 specifically for this project.

1. Values determined during Round 1. All other values determined during Rounds 2 and 3 of the Method Validation Study.

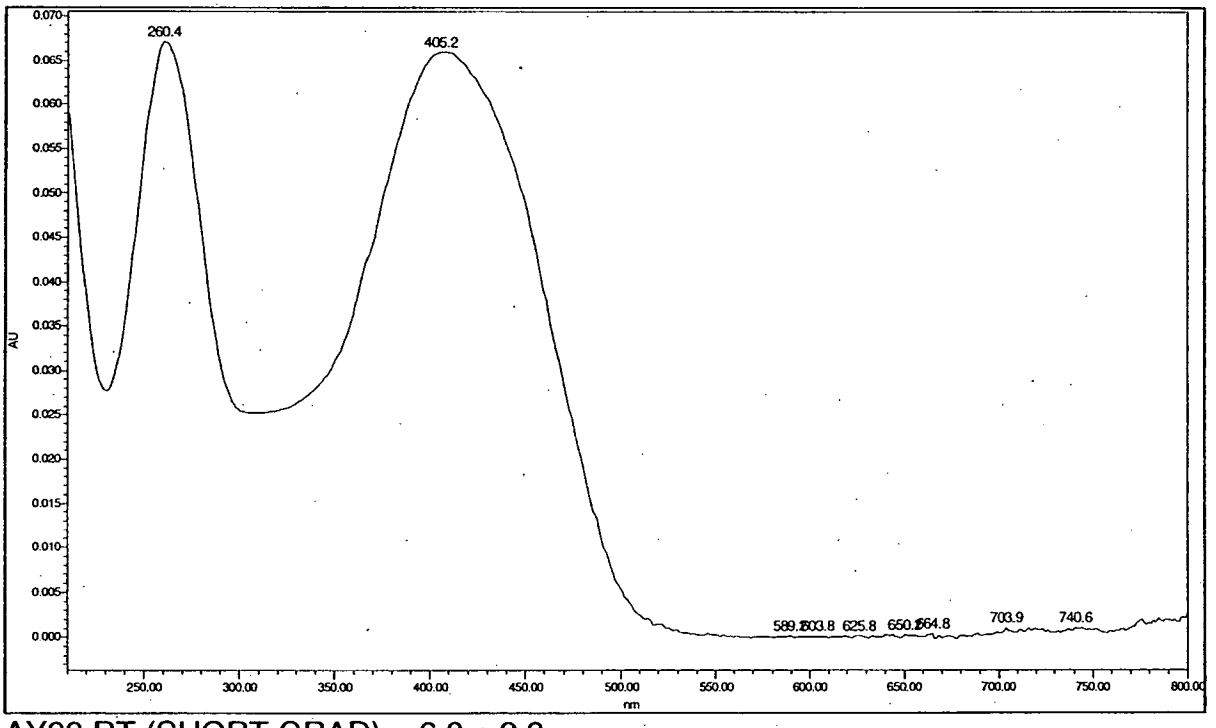
Table 5
Example Standard Concentrations for the Initial Calibration

Dye	Level 5 (ug/mL)	Level 4 (ug/mL)	Level 3 (ug/mL)	Level 2 (ug/mL)	Level 1 (ug/mL)
Acid Blue 45	37.200	18.600	9.300	4.650	2.325
Acid Yellow 23	40.200	20.100	10.050	5.025	2.513
Acid Yellow 73	36.200	18.100	9.050	4.525	2.263
Acid Orange 10	40.200	20.100	10.050	5.025	2.513
Acid Blue 1	42.600	21.300	10.650	5.325	2.663
Basic Yellow 2	37.000	18.500	9.250	4.625	2.313
Disperse Violet 1	26.020	13.010	6.505	3.253	1.626
Solvent Yellow 33	27.960	13.980	6.990	3.495	1.748
Disperse Blue 14	29.900	14.950	7.475	3.738	1.869
Acid Red 64	50.800	40.640	25.400	12.700	6.250
Acid Blue 9	36.200	18.100	9.050	4.525	2.263
Basic Violet 10	31.800	15.900	7.950	3.975	1.988
2-Aminoanthraquinone	37.380	18.690	9.345	4.673	2.336
Solvent Orange 3	25.580	12.790	6.395	3.198	1.599
1-Aminoanthraquinone	25.700	12.850	6.425	3.213	1.606
Solvent Yellow 14	28.320	14.160	7.080	3.540	1.770
Solvent Orange 7	28.800	14.400	7.200	3.600	1.800
Acid Yellow 3	36.600	18.300	9.150	4.575	2.288
Solvent Yellow 3	30.200	15.100	7.550	3.775	1.888
Benzanthrone	24.320	12.160	6.080	3.040	1.520
Anthracene	41.540	20.770	10.385	5.193	2.596
Solvent Red 1	46.380	23.190	11.595	5.798	2.899
Disperse Red 9	26.420	13.211	6.605	3.303	1.651
Solvent Yellow 2	31.920	15.960	7.980	3.990	1.995
Solvent Green 3	29.400	14.700	7.350	3.675	1.838
Solvent Red 24	5.680	2.840	1.420	1.136	0.710

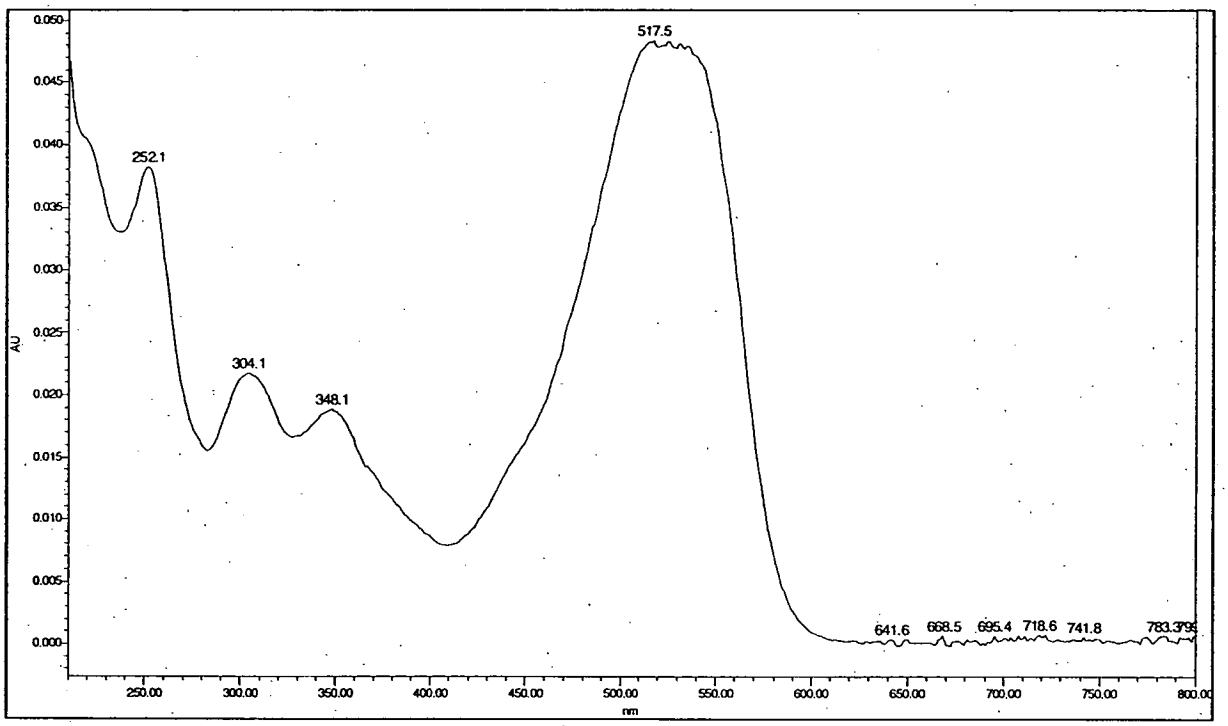
ATTACHMENT 1: SPECTRA AND APPROXIMATE RETENTION TIMES



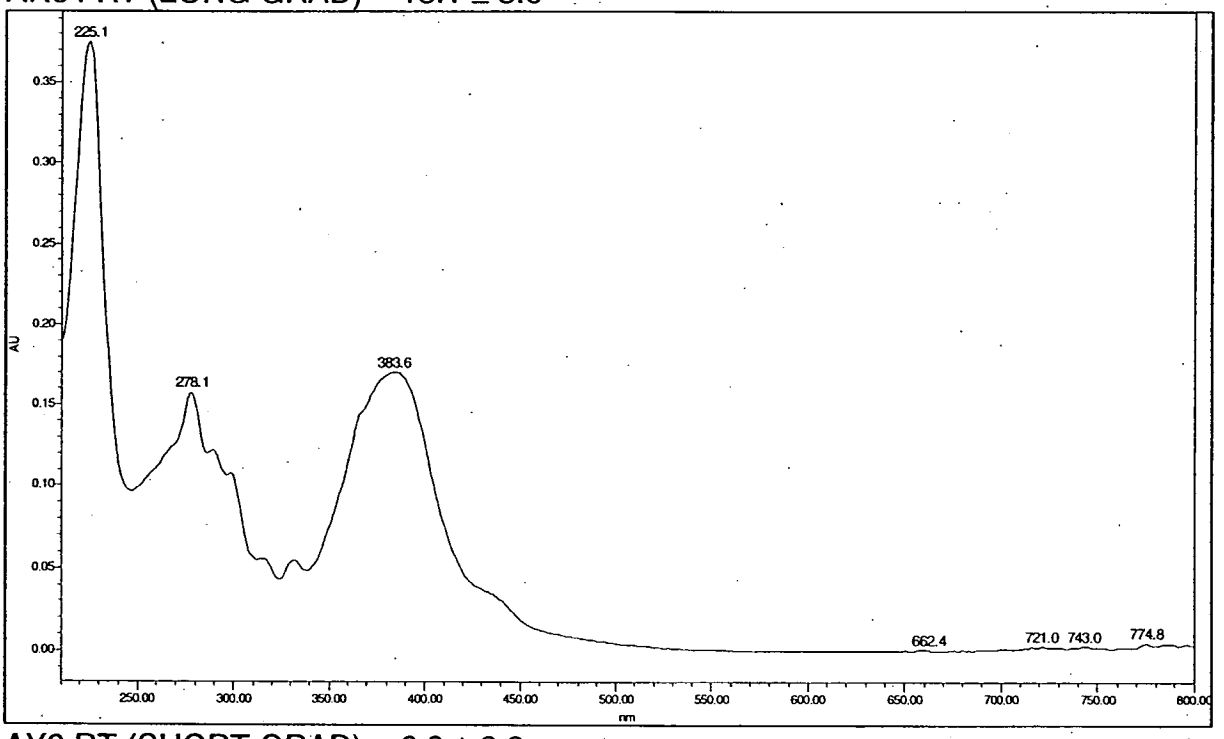
AB45 RT (SHORT GRAD) = 4.4 ± 2.0
AB45 RT (LONG GRAD) = 6.8 ± 10.6



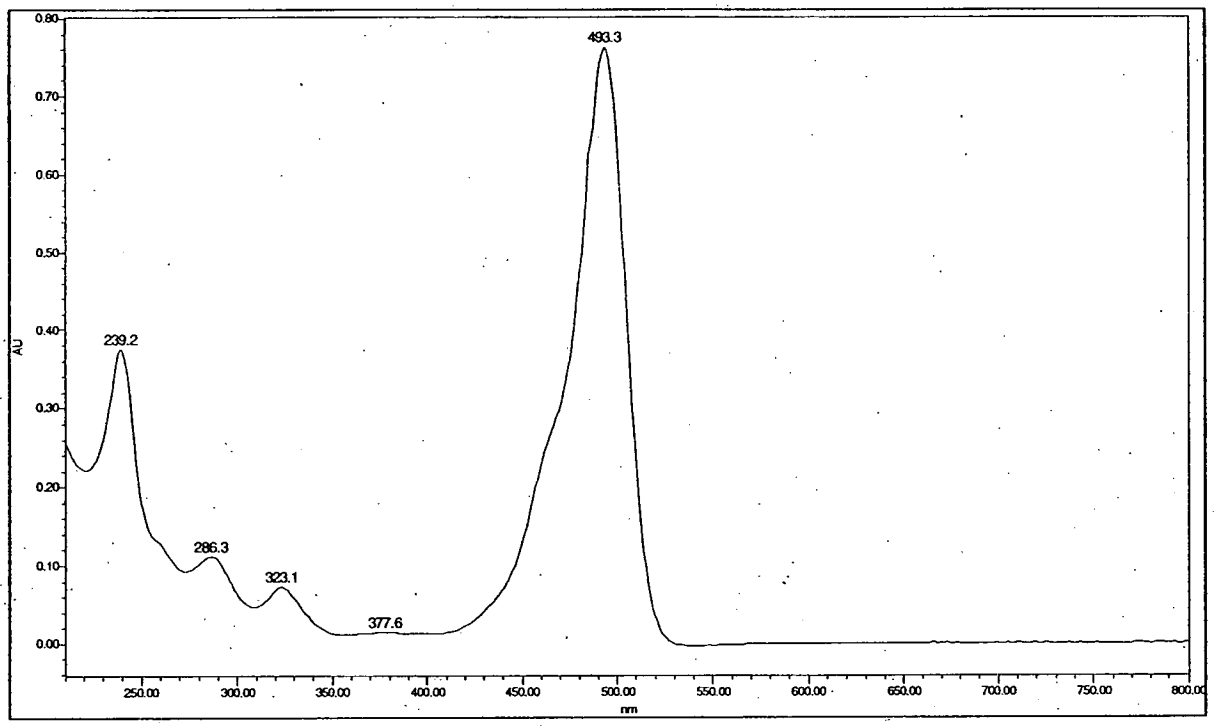
AY23 RT (SHORT GRAD) = 6.8 ± 3.3
AY23 RT (LONG GRAD) = 14.1 ± 5.2



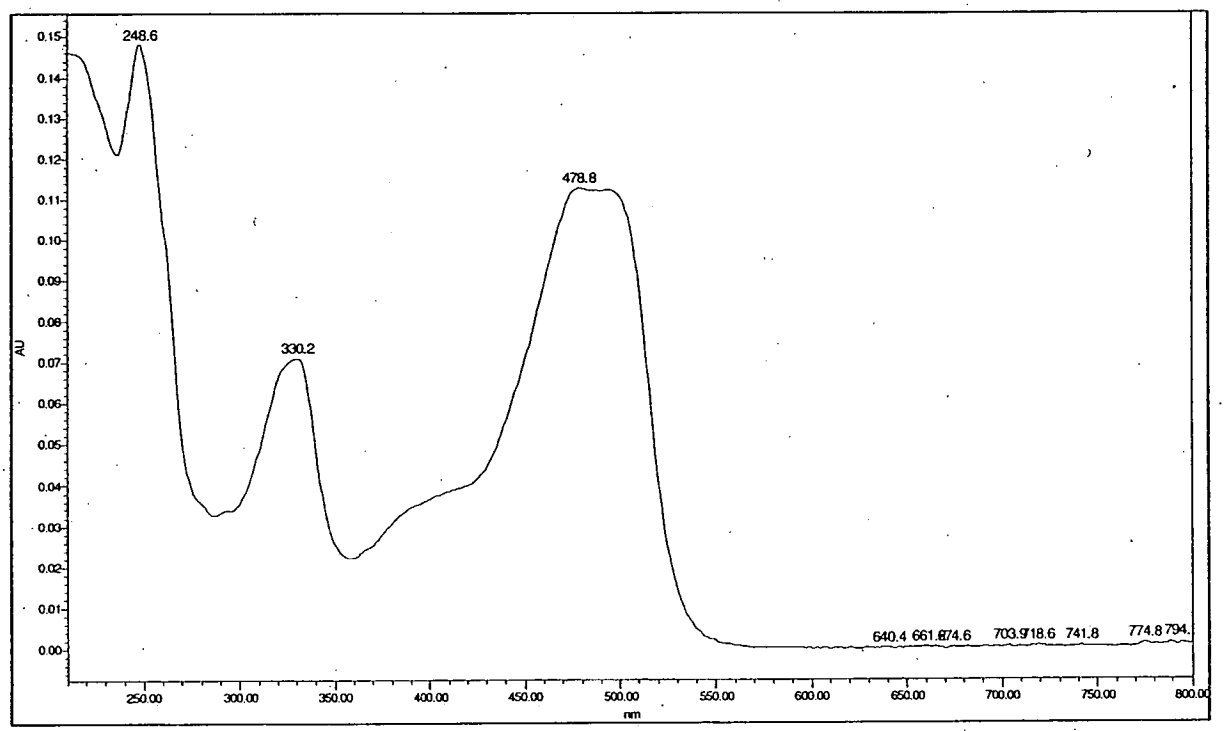
AR64 RT (SHORT GRAD) = 6.2 ± 2.7
AR64 RT (LONG GRAD) = 13.1 ± 3.0



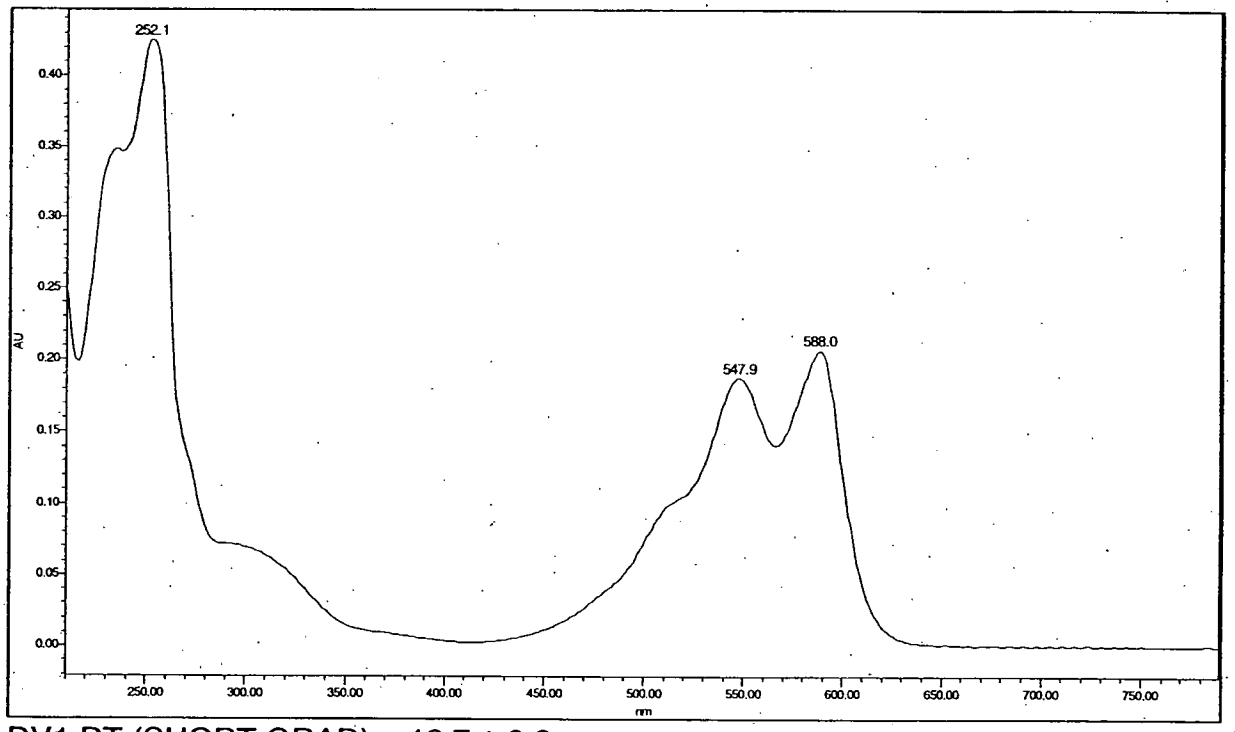
AY3 RT (SHORT GRAD) = 6.8 ± 2.9
AY3 RT (LONG GRAD) = 13.7 ± 3.4



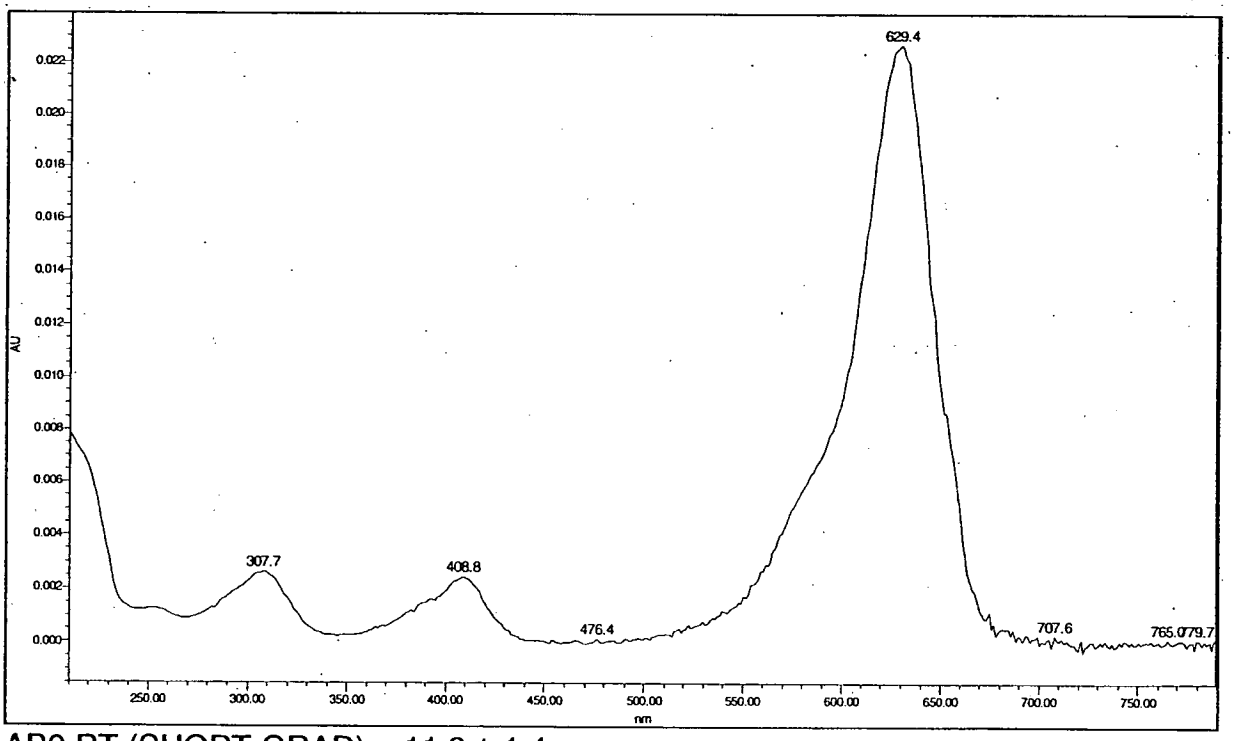
AY73 RT (SHORT GRAD) = 8.6 ± 2.3
AY73 RT (LONG GRAD) = 17.0 ± 4.0



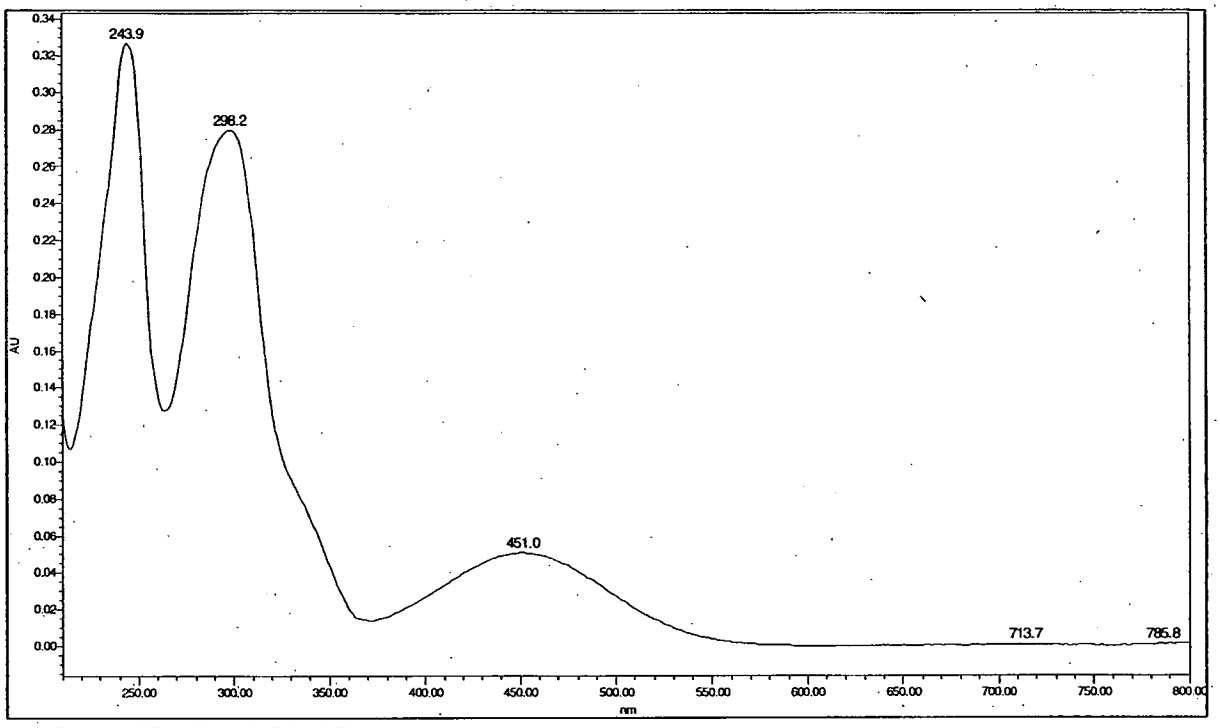
AO10 RT (SHORT GRAD) = 9.5 ± 2.1
AO10 RT (LONG GRAD) = 18.4 ± 3.6



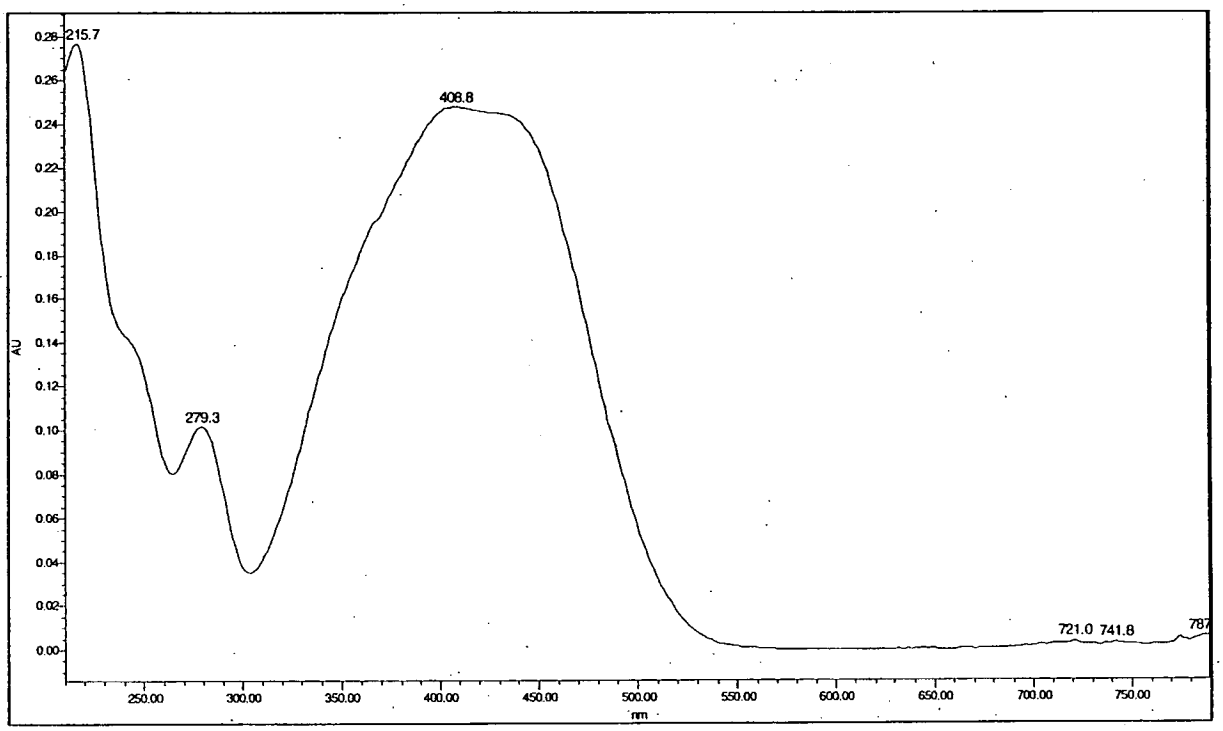
DV1 RT (SHORT GRAD) = 12.7 ± 0.2
DV1 RT (LONG GRAD) = 22.8 ± 0.2



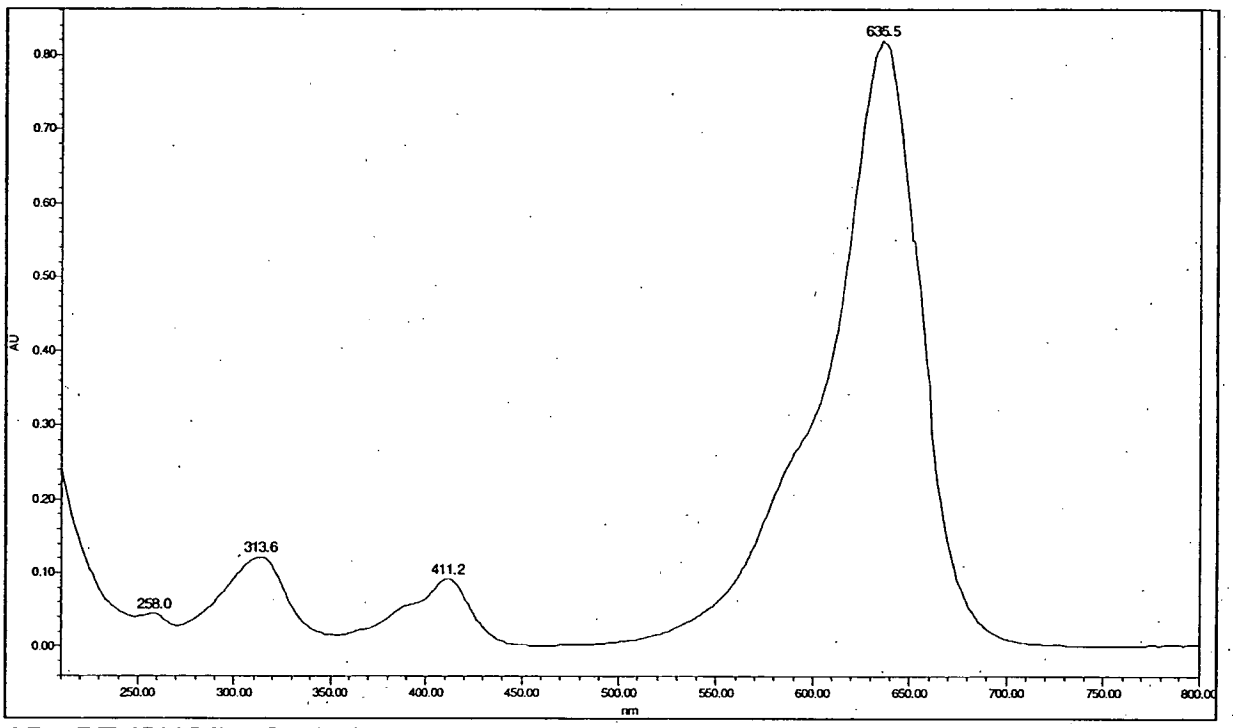
AB9 RT (SHORT GRAD) = 11.3 ± 1.4
AB9 RT (LONG GRAD) = 21.8 ± 2.6



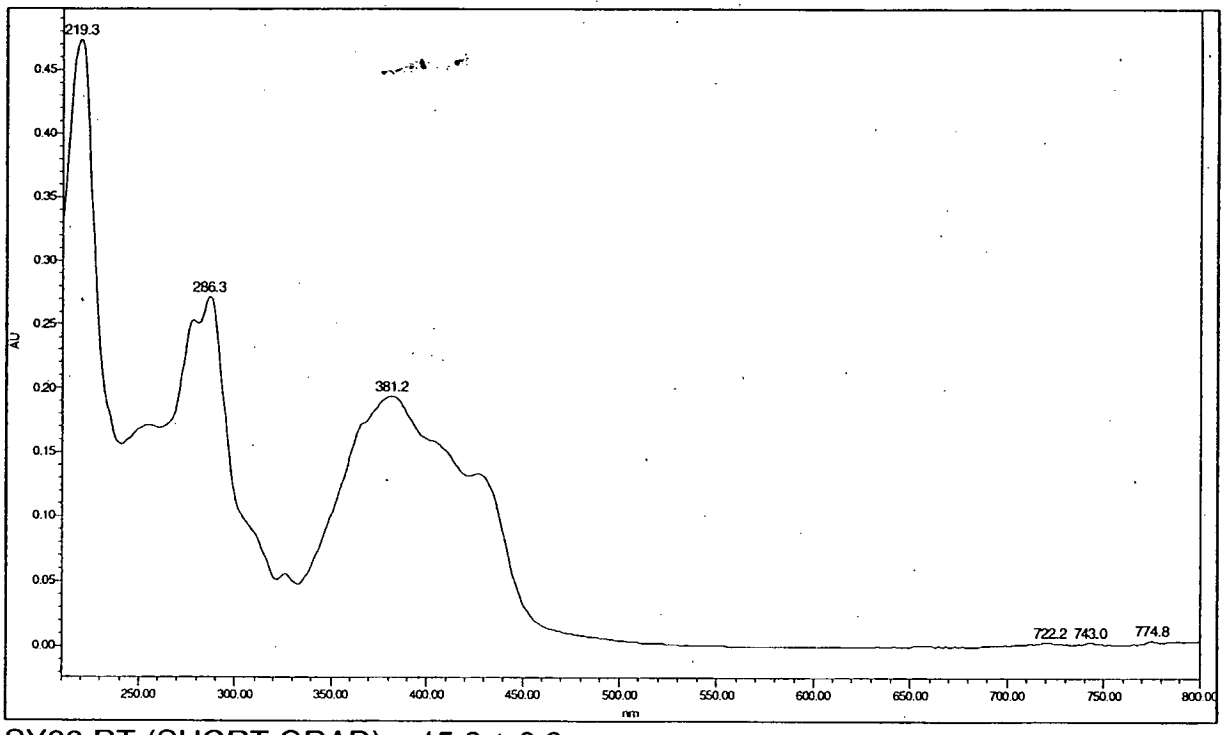
2AQ RT (SHORT GRAD) = 13.2 ± 0.5
2AQ RT (LONG GRAD) = 23.8 ± 0.5



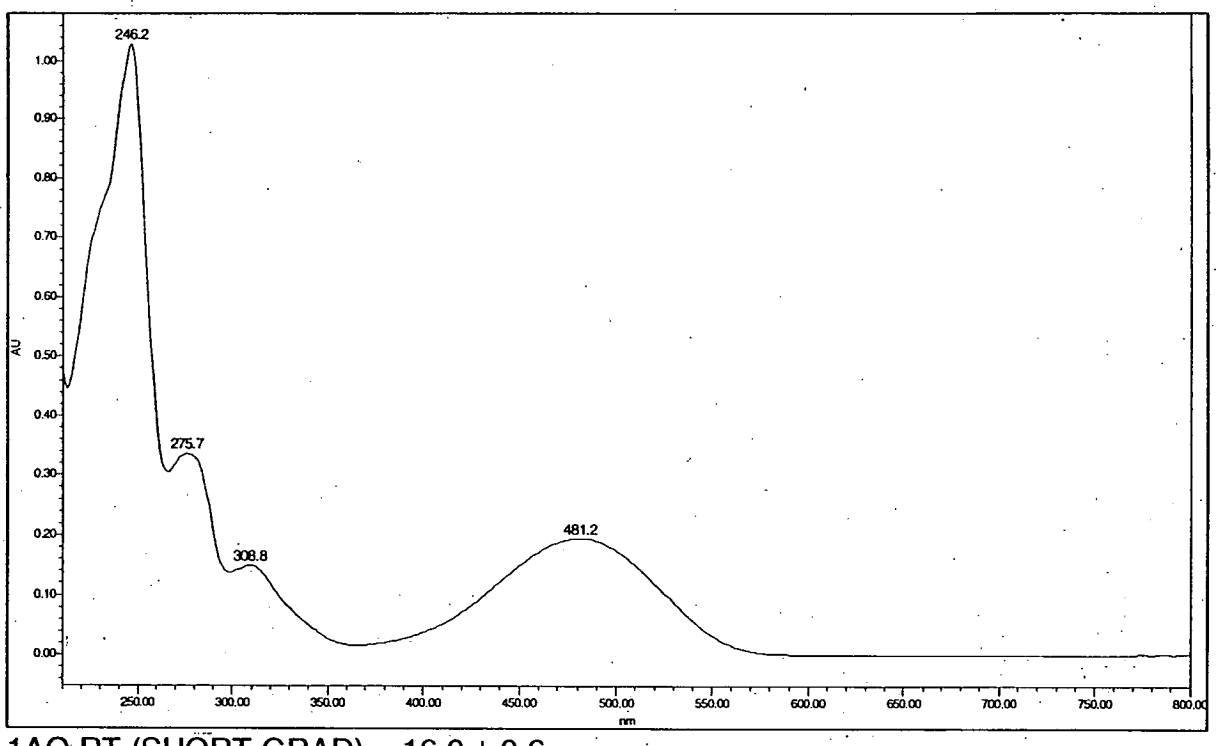
SO3 RT (SHORT GRAD) = 13.8 ± 0.2
SO3 RT (LONG GRAD) = 24.8 ± 0.2



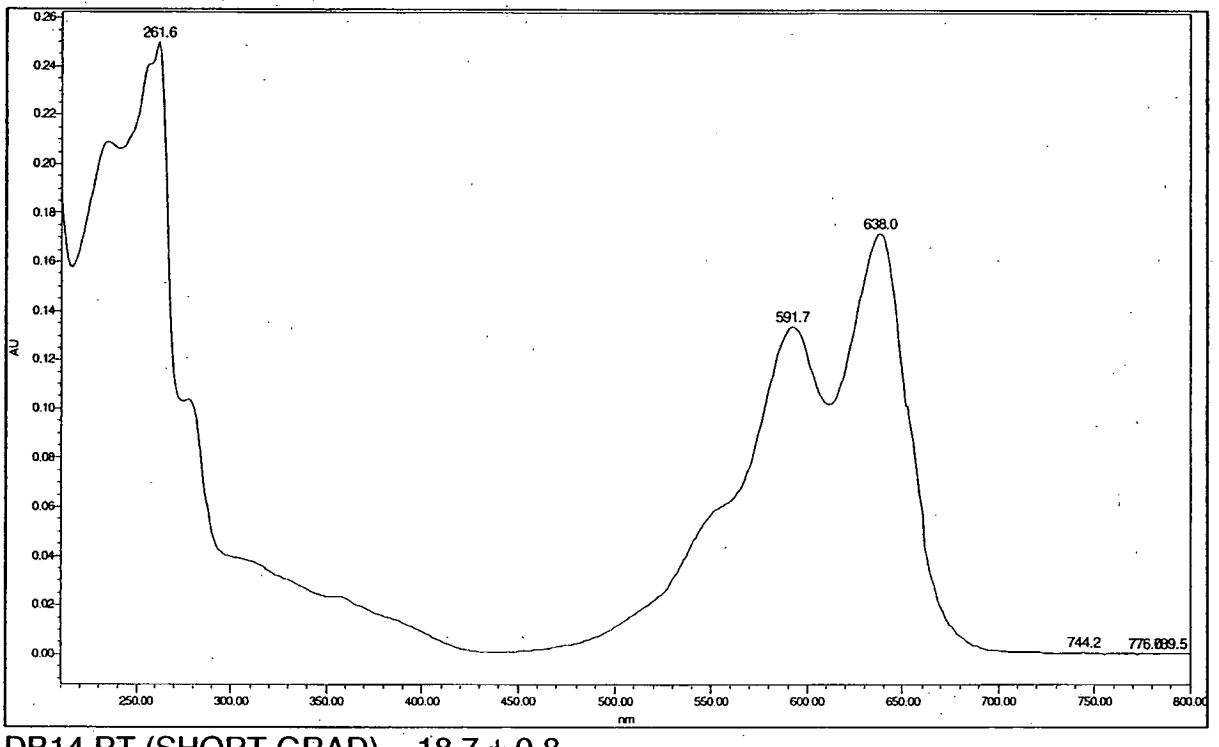
AB1 RT (SHORT GRAD) = 12.4 ± 1.5
AB1 RT (LONG GRAD) = 23.5 ± 2.3



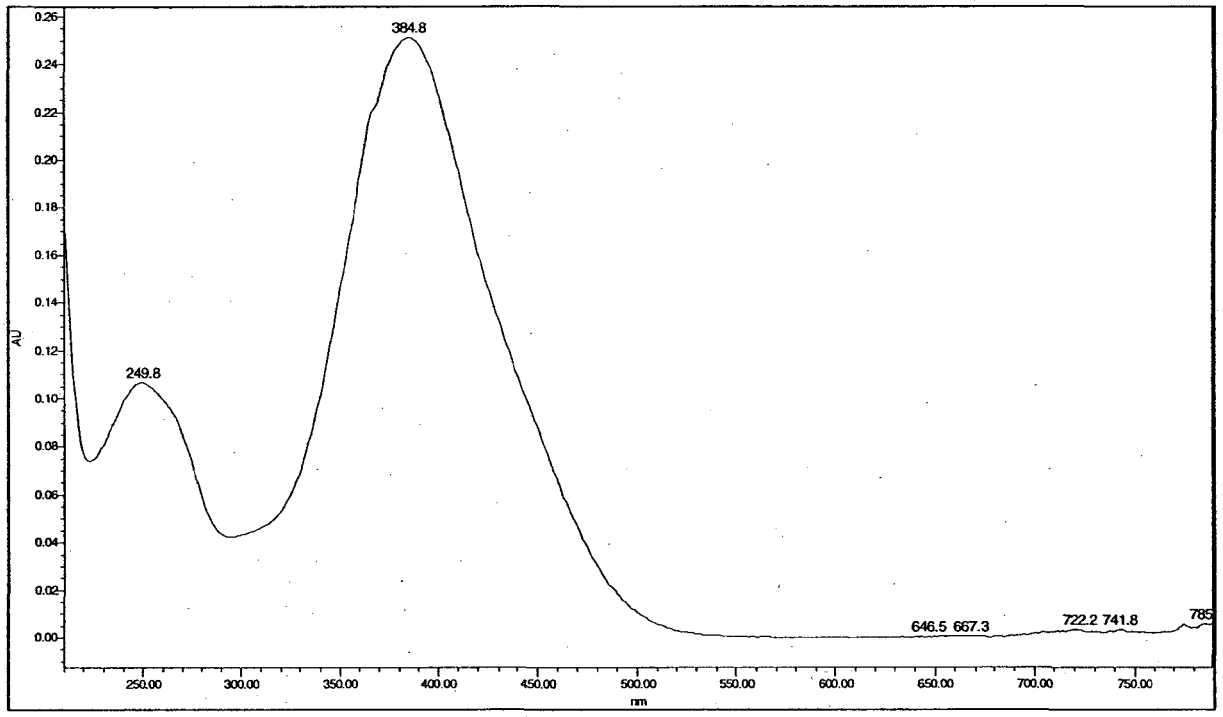
SY33 RT (SHORT GRAD) = 15.3 ± 0.8
SY33 RT (LONG GRAD) = 28.1 ± 1.2



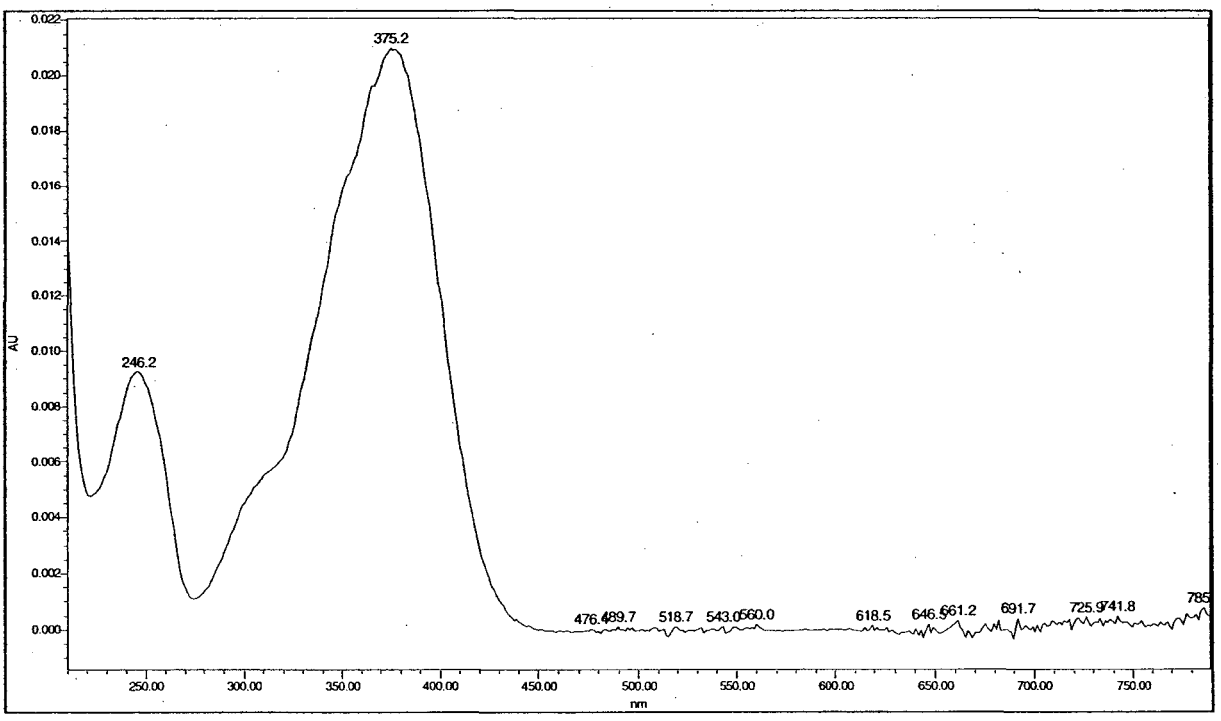
1AQ RT (SHORT GRAD) = 16.0 ± 0.6
1AQ RT (LONG GRAD) = 28.9 ± 0.9



DB14 RT (SHORT GRAD) = 18.7 ± 0.8
DB14 RT (LONG GRAD) = 33.9 ± 1.0



SY3 RT (SHORT GRAD) = 19.8 ± 0.7
SY3 RT (LONG GRAD) = 36.1 ± 1.0



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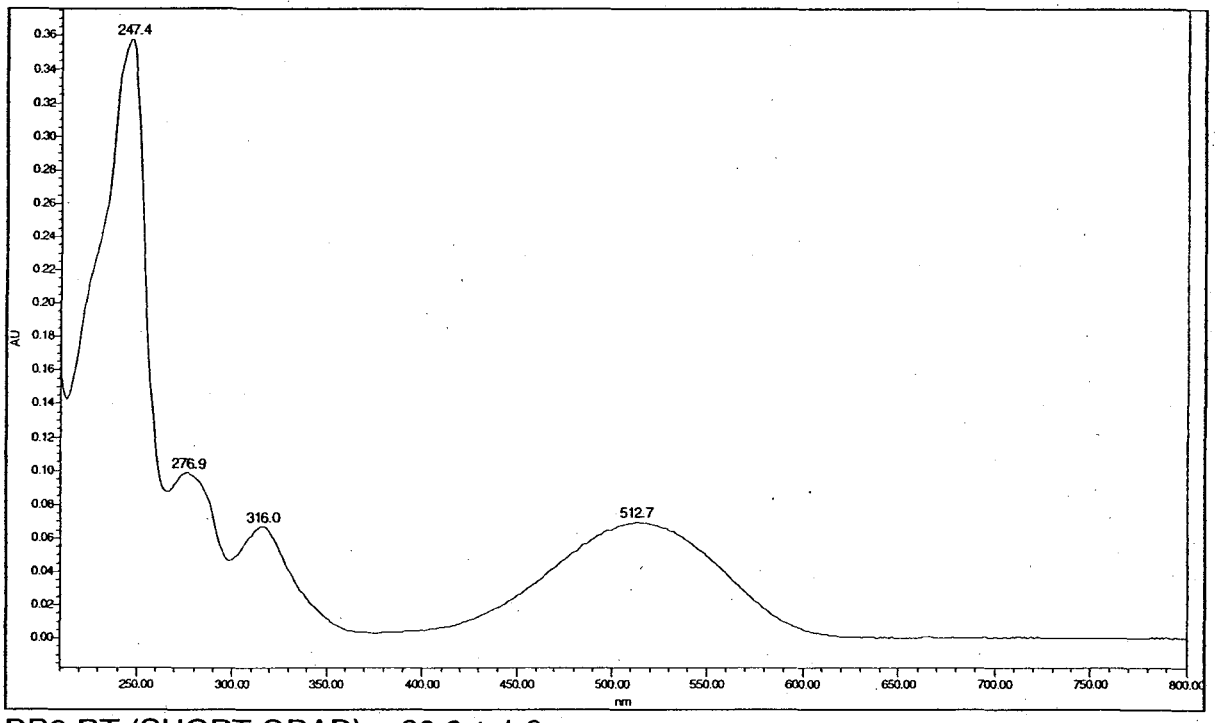
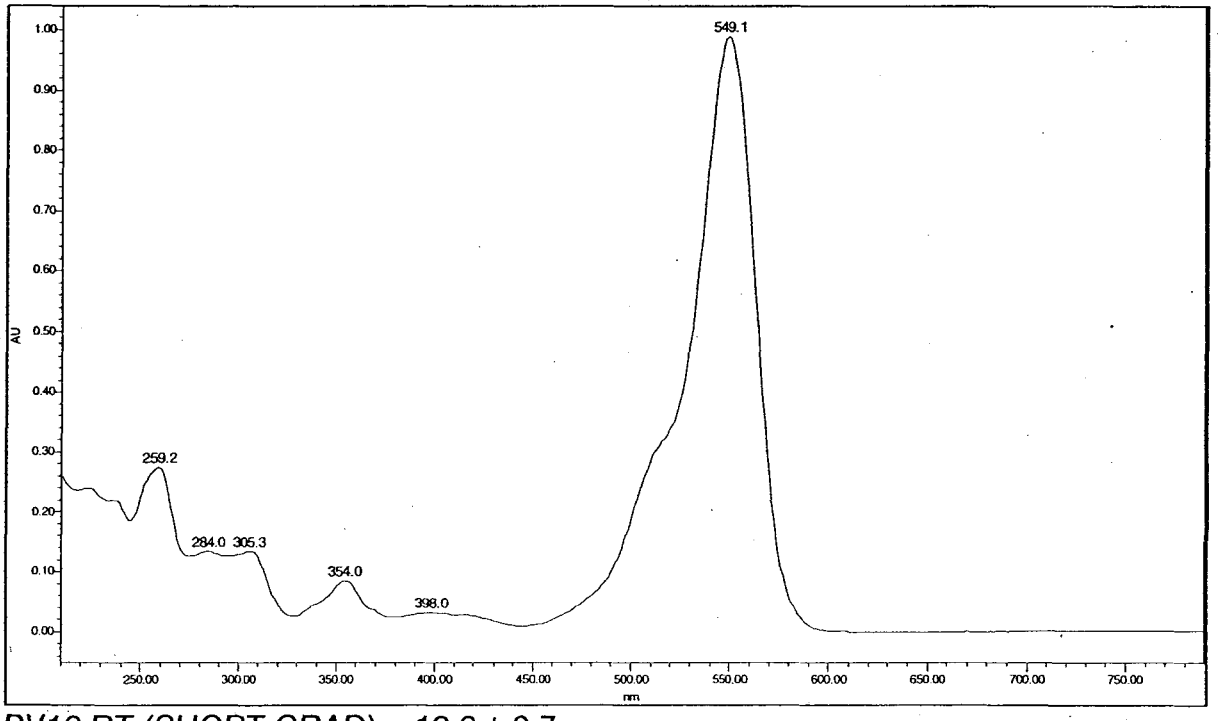
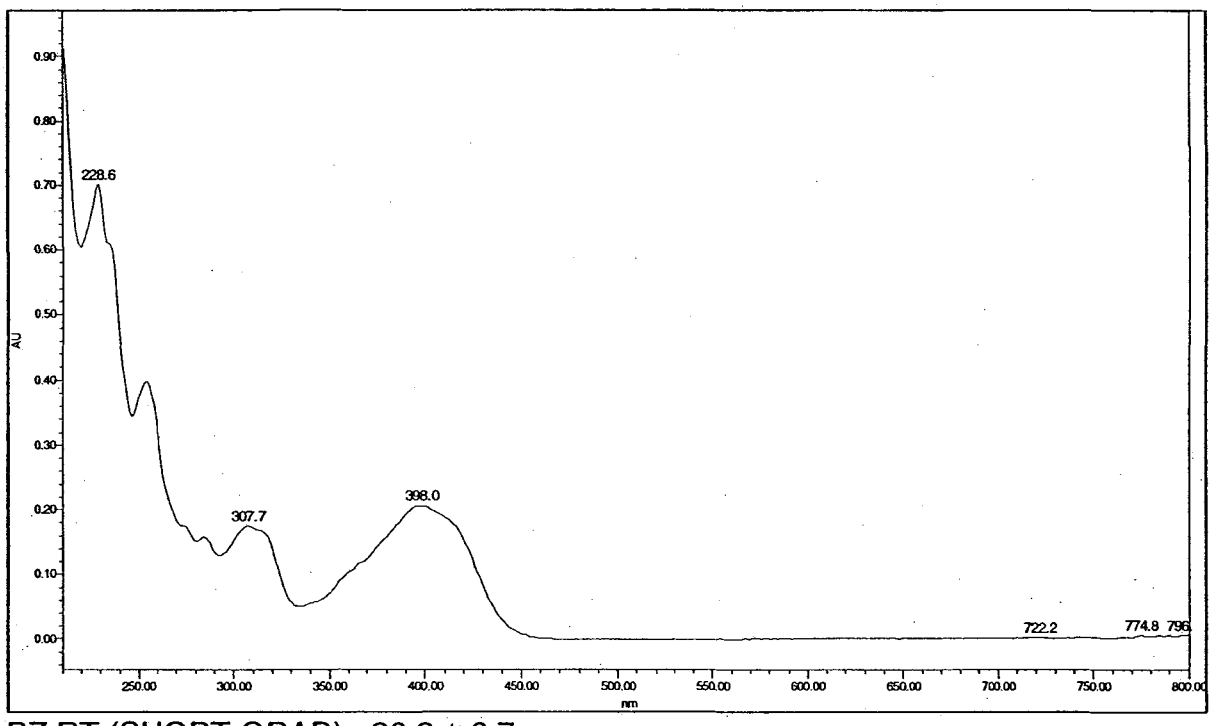
BY2 RT (LONG GRAD) = 35.2 ± 1.0



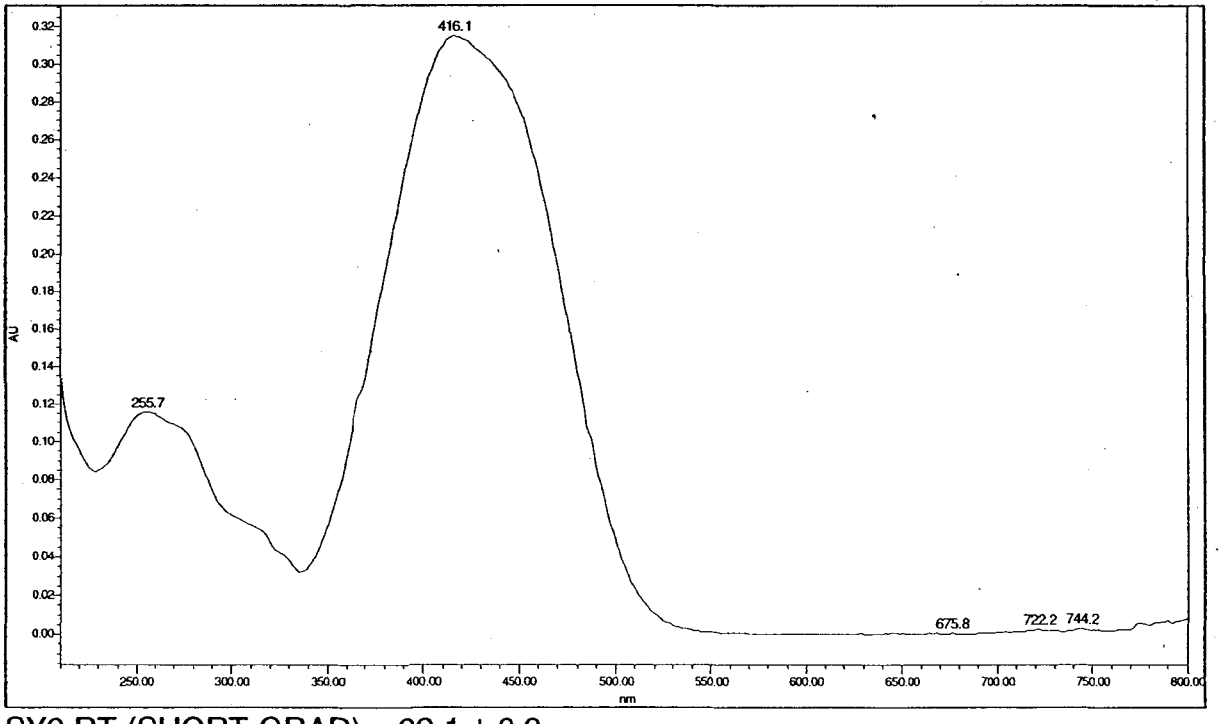
DR9 RT (SHORT GRAD) = 20.2 ± 1.0
DR9 RT (LONG GRAD) = 36.5 ± 1.5



BV10 RT (SHORT GRAD) = 19.6 ± 0.7
BV10 RT (LONG GRAD) = 36.0 ± 0.8



BZ RT (SHORT GRAD) =20.3 ± 0.7
BZ RT (LONG GRAD) = 36.8 ± 0.9



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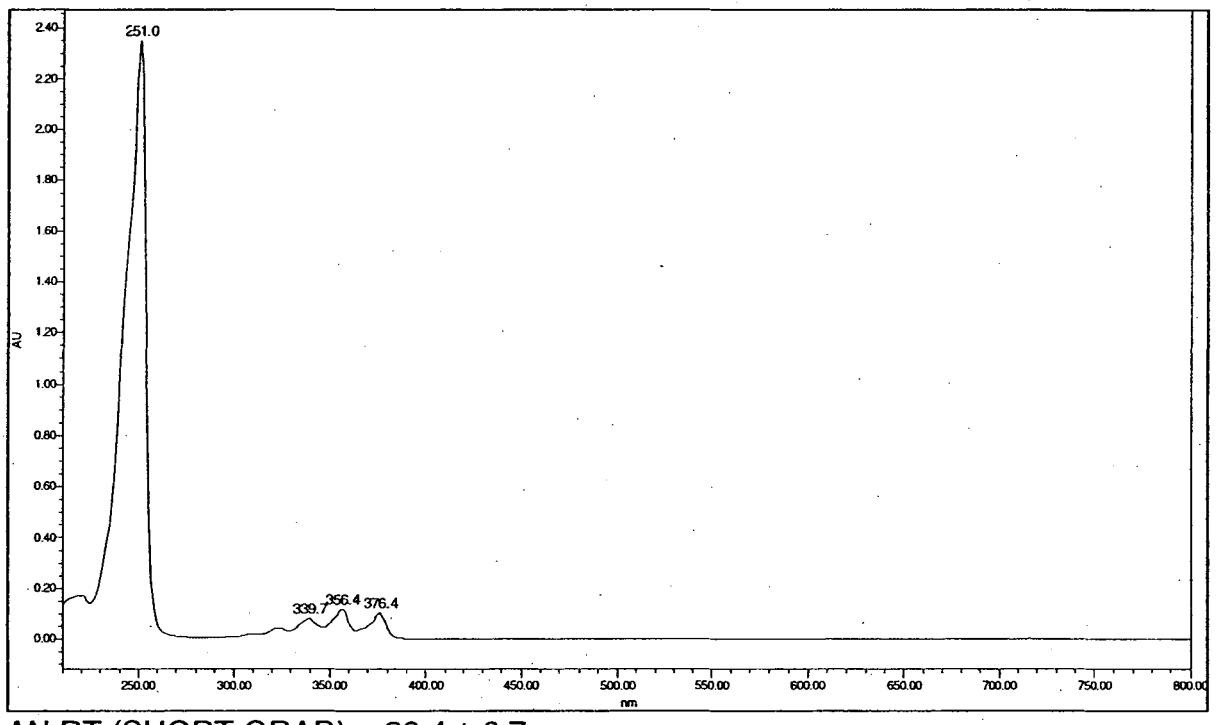
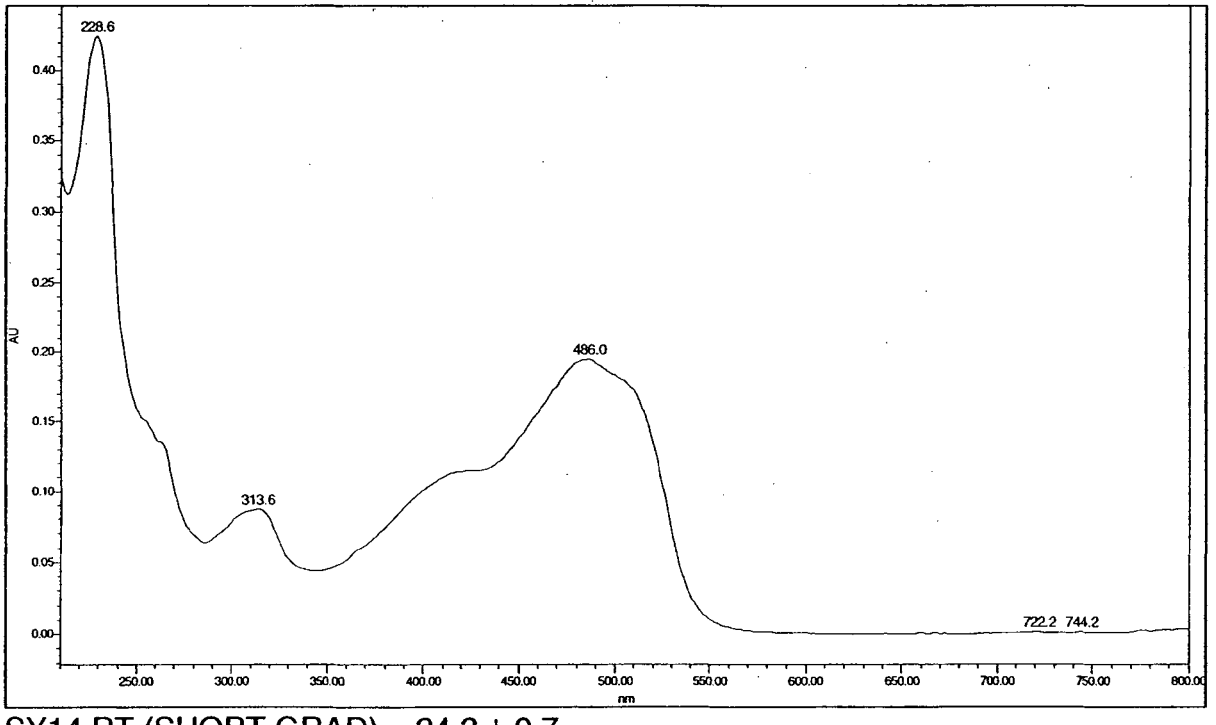
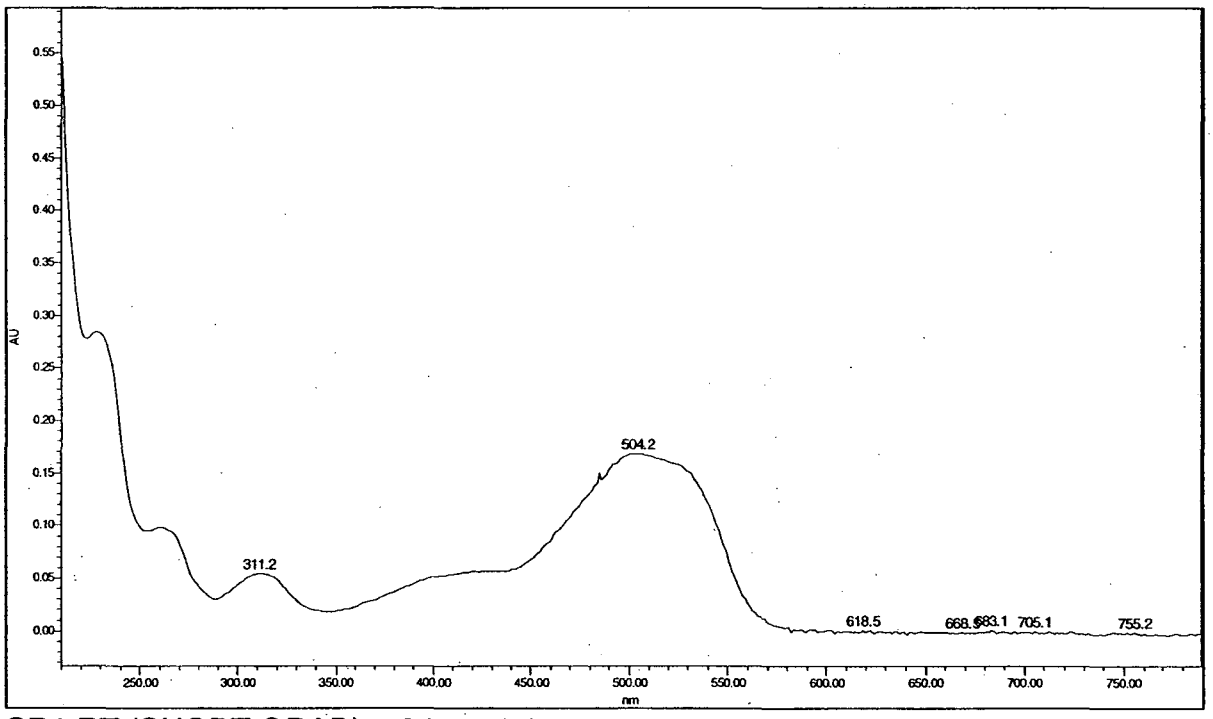
SY2 RT (LONG GRAD) = 40.2 ± 1.4



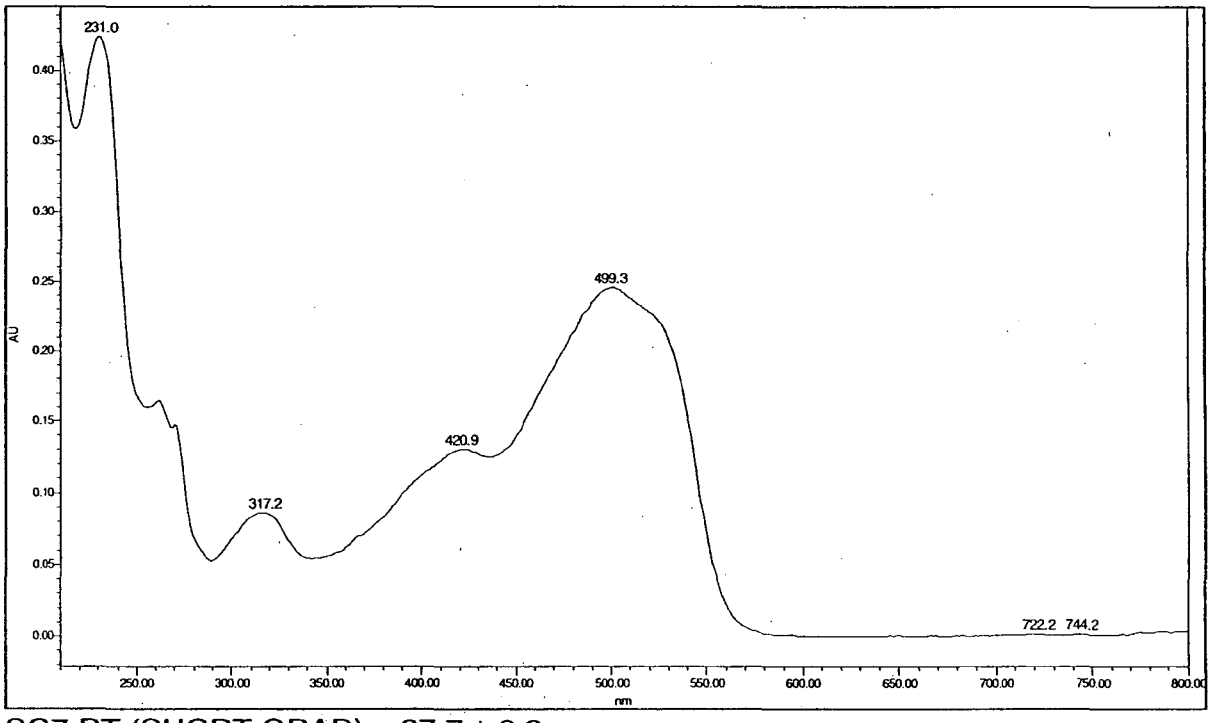
AN RT (SHORT GRAD) = 22.4 ± 0.7
AN RT (LONG GRAD) = 40.7 ± 1.1



SY14 RT (SHORT GRAD) = 24.3 ± 0.7
SY14 RT (LONG GRAD) = 44.4 ± 1.3



SR1 RT (SHORT GRAD) = 24.5 ± 0.8
SR1 RT (LONG GRAD) = 44.9 ± 1.1



SO7 RT (SHORT GRAD) = 27.7 ± 0.6
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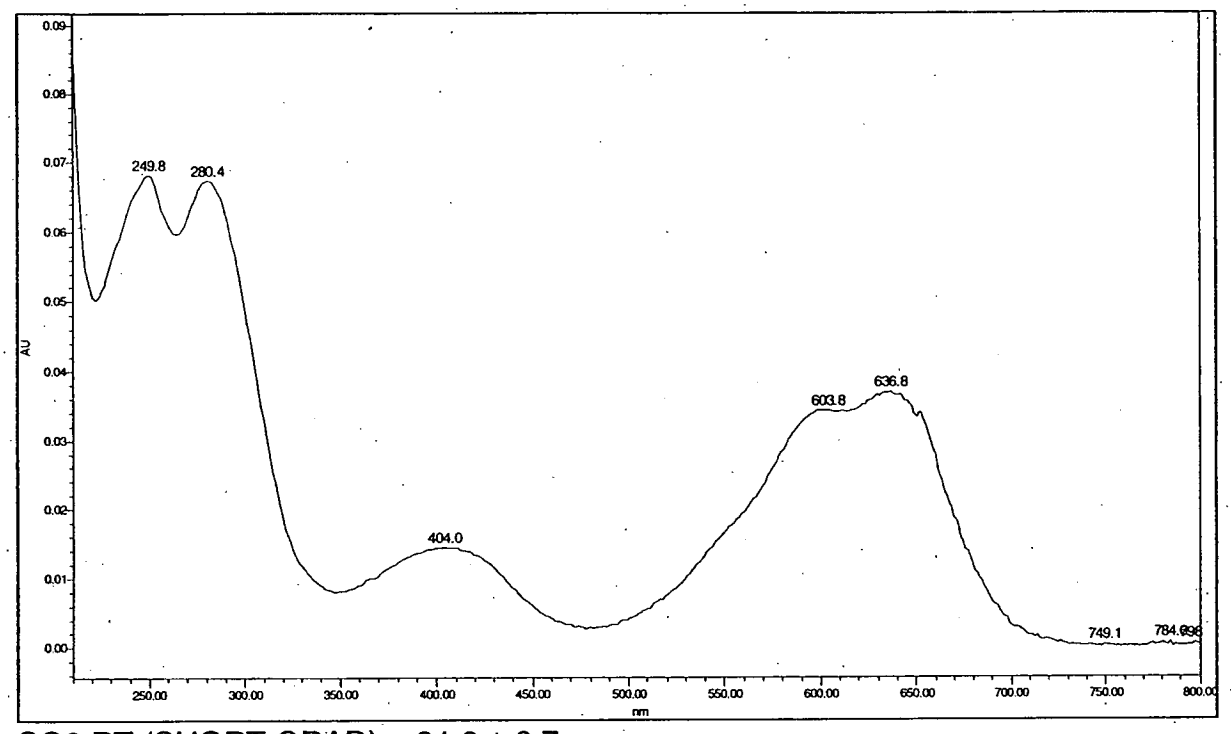
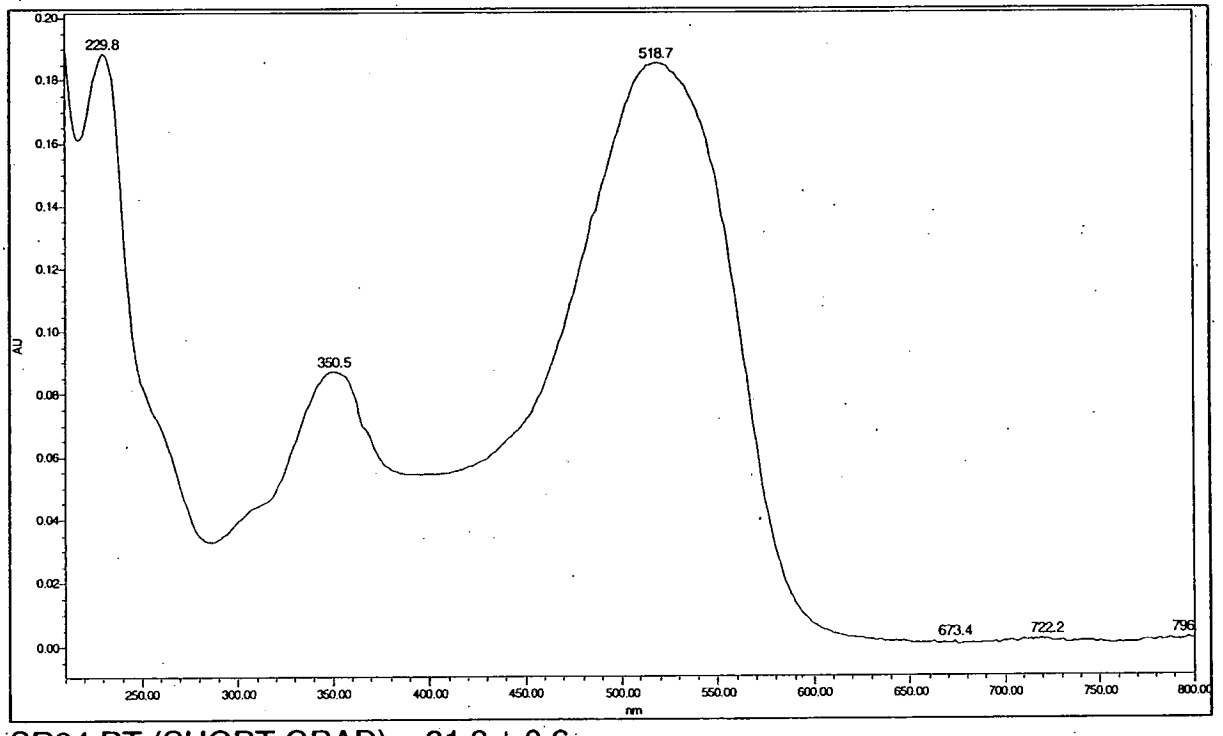


SG3 RT (SHORT GRAD) = 31.0 ± 0.7
SG3 RT (LONG GRAD) = 57.8 ± 1.3



SR24 RT (SHORT GRAD) = 31.3 ± 0.6
SR24 RT (LONG GRAD) = 58.2 ± 1.0

TED/C-405-3001A

ORDNANCE ENGINEERING DIRECTORATE
CRANE DIVISION
NAVAL SURFACE WARFARE CENTER
Crane, Indiana 47522-5001

DIRECTIVE

FOR

CONTROL OF TECHNICAL DOCUMENTS AND ENGINEERING DRAWINGS

MASTER

NAVAL SEA SYSTEMS COMMAND

NSWC

Naval Surface Warfare Center

CRANE DIVISION

CR-405-3001A

[redacted]

APPROVAL

DIRECTIVE
for
CONTROL OF TECHNICAL DOCUMENTS AND ENGINEERING DRAWINGS

CR-405-3001 Rev A

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Prepared By — Date
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RECORD OF CHANGES

DIRECTIVE
for
CONTROL OF TECHNICAL DOCUMENTS AND ENGINEERING DRAWINGS

CHANGE	DATE OF CHANGE	TITLE OR BRIEF DESCRIPTION	ENTERED BY

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1.0 PURPOSE AND SCOPE

1.1 PURPOSE. To ensure all technical documents used by the Test and Evaluation Department, Ordnance Engineering Directorate at Naval Surface Warfare Center, Crane Division, are complete, accurate, current, controlled and distributed to the appropriate personnel, and can be readily retrieved.

1.2 SCOPE. Applies to the approval, issue, storage, revision, and recall of all technical documents, including engineering design drawings (CAD or manual) and Standard Operating Procedures (SOPs). Applies to all personnel within the Test and Evaluation Department who submit technical documents to Documentation Repository 08 for document control.

2.0 DEFINITIONS

2.1 CHANGES; Minor revisions which do not effect the scope, policy, assignment of responsibilities, and do not significantly revise administrative or step-by-step procedures.

2.2 CONTROL; Implies that documents are received, maintained, inventoried and issued by Repository 08.

2.3 CONTROLLED DOCUMENTS; Copies of externally or internally generated technical documents designated for formal control by a documentation repository. All controlled documents issued from the repository shall be identified with a stamp in RED ink indicating that it is a "Controlled Copy" showing the repository number from which issued and the copy number. Holders of controlled copies are issued updates as they are received.

2.4 DOCUMENTATION REPOSITORY; A function, established by directorates, that receives documents, maintains inventories, issues copies and disposes of obsolete documents. Documentation repositories are managed by authorized document control custodians.

2.5 ENGINEERING DRAWING; An original drawing prepared by/for an individual in the Test and Evaluation Department to be used for procurement, or one that defines any fixture, equipment or system used to perform tests or evaluations on a product.

2.6 MASTER; An original or principle document submitted to Documentation Repository 08 for control. The source document from which copies are reproduced.

2.7 REVISIONS; A change or accumulation of changes that affects 50% or more of all pages in the document.

2.8 TECHNICAL DOCUMENT; Drawings, procedures, work instructions and specifications that govern the design and development, manufacturing, maintenance and operational use of a product or service, or the process (such as test and evaluation) that provides the product or service.

2.9 UNCONTROLLED DOCUMENTS; Copies of technical documents which are information only copies and are not to be used in the performance of work tasks. These copies shall be stamped in red ink "FOR INFORMATION ONLY" or "UNCONTROLLED COPY". These copies will not be updated and no record of who has Uncontrolled Documents will be maintained.

3.0 REQUIREMENTS

3.1 DOCUMENT CONTROL

3.1.1 All documents to be controlled shall be given to the document control custodian via electronic means (on disk or e-mailed), or if electronic means are not available, hard copy.

3.1.2 The custodian will then enter the document into the document management software for storage and routing. Hardcopies of new documents not in electronic form shall be scanned into the system when practical. It is the responsibility of the document preparer to furnish the custodian with a list of personnel for routing the document. The draft copies shall be returned to the preparer for review and implementation of any changes and then rerouted by the preparer for final approval.

3.1.3 Once the document has been routed and approved, the resulting document will be permanently stored in the document control system. The document control system will be the only approved source for duplicate copies of the document. All controlled technical documents will be stored on the network server in electronic form or in a designated area (noted in document control system) in hardcopy. Only the Document Control Custodian and alternate (or Contractor Support) will be able to input documents into the electronic document repository.

3.1.4 The document control custodian shall maintain a master list of all controlled technical documents and their revision status and shall be able to generate the list upon request. The document control custodian shall also be able to generate a master list by project number and/or code.

3.1.5 Personnel requesting a hard copy of a document shall first receive authorization from the preparer, project manager, or organizational manager. The custodian will then issue a copy with the front page stamped in RED ink identifying that the copy is controlled, the repository number and the unique copy number. Records of controlled documents issued shall be maintained by the document control custodian. All other copies shall be considered uncontrolled and unofficial and shall be used only as reference material. Any copy that is uncontrolled shall be stamped "FOR INFORMATION ONLY" or "UNCONTROLLED COPY".

3.1.6 When only a section of a document is required, a list of sections and respective users shall be provided to the Document Control Custodian. The Custodian shall issue controlled sections, following the same procedures for issuing controlled documents, except that each page of a controlled section shall be stamped and controlled. The controlled section information shall be stored with the master documents.

3.1.7 The document control custodian shall use the Document Receipt Form (9ND-NWSCC 4430/10) when issuing a hard copy controlled copy of a technical document. Recipients of controlled documents shall acknowledge receipt by signing and returning to the issuing custodian the yellow-card portion of the Document Receipt Form within five working days of the receipt. The document control custodian shall reconcile all Document Receipt Forms issued on a monthly basis and resolve all discrepancies.

3.2 CHANGES/REVISIONS

3.2.1 The original preparer of the controlled document, when possible and practical, shall review and approve changes/revisions, and shall note the changes in the document or on an attachment before routing for approval.

3.2.2 When final approved changes or revisions of controlled documents are received, the document control custodian shall request all controlled copies of the superseded document be returned, using the Document Receipt Form (9ND-NWSCC 4430/10). When returned, it shall be noted in the record. If the controlled copy of a document cannot be located, the responsible person shall forward a written statement to the custodian stating this.

3.2.3 Superseded and cancelled Master documents that have been document controlled shall be retained on the network server for historical purposes and shall be identified as such. Superseded and cancelled Master hard copies shall be physically retained, with the location noted in the document control system.

3.3 CLASSIFIED MATERIAL

3.3.1 Any classified technical documents shall be stored in a designated locked cabinet/safe. Classified documents as a whole will not be scanned or stored on the network server; only the unclassified information such as titles and subjects will be entered into the document control system. Along with the title and subject, location of the actual document shall be noted for inventory/master list purposes.

3.3.2 Classified documents shall be physically inventoried once a year by the document control custodian.

4.0 RESPONSIBILITY

4.1 DEPARTMENT MANAGER

4.1.1 Ensure current and appropriate documents are available at all locations where operations take place.

4.1.2 Establish documentation repositories, as required.

4.1.3 Appoint a Documentation Repository Custodian and alternate for each repository.

4.1.4 Ensure this policy is available and followed by all Department Personnel.

4.1.5 Approve Project/Organizational Managers personnel listings for reading/writing access of the electronic document control repository.

4.2 DOCUMENT RECIPIENT/USERS

4.2.1 Acknowledge receipt of controlled documents.

4.2.2 Return of obsolete controlled documents, as directed by the issuing custodian, within five working days.

4.2.3 When controlled documents are no longer needed, users shall return them to the issuing repository, unless other arrangements are made with the custodian.

4.2.4 Inform the custodian of any changes in organization, code or location.

4.2.5 Ensure current and appropriate documents are available at all locations where operations take place.

4.3 DOCUMENT REPOSITORY CUSTODIAN

4.3.1 Maintain and control all technical documents submitted by department personnel.

4.3.2 Maintain and control/limit access to both hard copy and electronic master documents.

4.3.3 Maintain a master list of technical documents controlled by the repository, to be produced upon request.

4.3.4 Introduce new documents into the document control system.

4.3.5 Route copies of electronically submitted documents for initial review/comments.

4.3.6 Issue controlled and uncontrolled copies of documents, as needed.

4.3.7 Report lost controlled documents by memorandum, via project manager or organizational manager, to the department manager as soon as it is apparent that documents are missing.

4.3.8 Maintain records for all controlled copies issued.

4.3.9 Maintain all superseded and obsolete documents for historical purposes.

4.3.10 Scan, as appropriate, controlled documents for storage if not received in electronic form. It will not be necessary at this time to scan old, unused documents or very large drawings. Management will decide when and if old seldom used documents will be scanned into the Document Control system or maintained in their current hard copy form.

4.3.11 Be subject to any external or internal audits performed.

4.3.12 Inventory all classified documents held by the department once a year.

4.3.13 Issue in-house department drawing numbers from the Drawing Number Log book.

4.3.14 Issue SOP numbers.

4.3.15 Ensure drawing number and title on the final drawing match the number assigned. Stamp hardcopy master documents Master. Store in designated area.

4.3.16 Stamp any old hard copy documents superseded.

4.4 PREPARERS

4.4.1 Prepare and maintain documents in accordance with Branch guidelines and this instruction.

4.4.2 Review and approve changes/revisions to documents originated.

4.4.3 State nature of change on document or on attachment.

4.4.4 Ensure that review copies of all new and revised technical documents are routed to pertinent personnel within and outside the Department.

4.4.5 Prepare and route electronic copies for final approval after initial routing for review/comments has been completed.

4.4.6 Ensure that the document control custodian receives notification of final approvals.

4.4.7 Route any hard copy documents needing approval.

4.5 PROCESS SUPERVISOR

4.5.1 Assure that only approved document controlled technical documents are available at the work site where operations are being performed.

4.5.2 Perform required document reviews, and note these reviews as required.

4.6 PROJECT ENGINEERS/TECHNICIANS

4.6.1 Inform the document control custodian of the number of controlled copies of technical documents required for testing, as well as how many uncontrolled copies, if any, are needed.

4.6.2 Assure all controlled copies of documents under their cognizance are returned to the document control custodian after they are no longer needed. Uncontrolled copies may also be returned to document control, but it is not a requirement.

4.6.3 Have drawings revised based on conformance with current individual branch instruction.

4.6.4 Ensure current and appropriate documents are available at all locations where operations take place.

4.7 PROJECT/ORGANIZATIONAL MANAGERS

4.7.1 Identify technical documents that require control.

4.7.2 Ensure that repository custodians receive all documents and changes/revisions for which control in requested.

4.7.3 Provide repository custodians with authorized distribution lists and update this list as needed.

4.7.4 Provide the applicable project number and/or organizational code for each new/revised document to be entered into the document control system.

4.7.5 Assure approved technical documents are prepared correctly. Assure final technical documents are placed in the document control system in the repository.

4.7.6 Ensure current and appropriate documents are available at all locations where operations take place.

4.7.7 Provide to Document Control Custodian, via the Department Manager, a listing of personnel permitted access to the electronic document repository for read and/or print access. Write access to the electronic document repository is limited to the Document Control Custodian, alternate and Document Control support personnel.

5.0 REFERENCE DOCUMENTS

5.1 NSWC DIVINST 8000.1 ACN 1; Standard Operating Procedures Program for the Processing of Expendable Ordnance

5.2 FBMPI 200D; Fleet Ballistic Missile Program Instruction

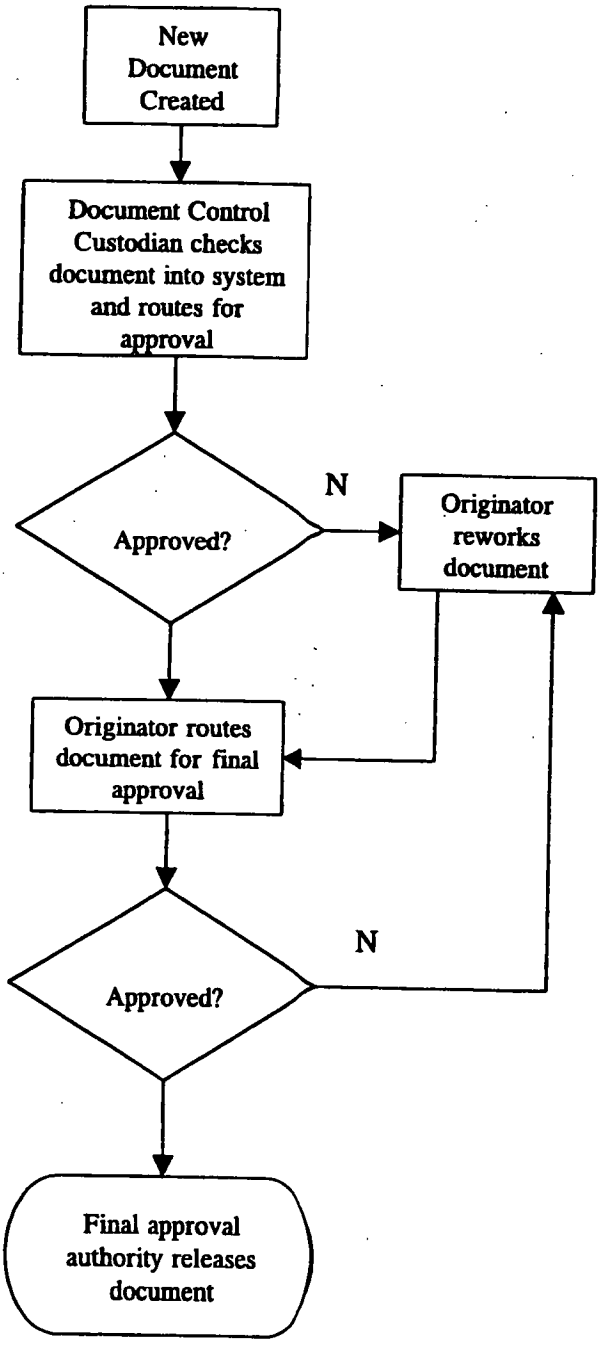
5.3 SMBD D-4056-1001 C-1; Instruction for Preparation, Use and Control of Strategic Missile Branch Directives

5.4 NWSCCINST 4355.34C; Handbook for Controlling Documentation

5.5 ISO 9000 INSTRUCTIONS Element 4.5; Document and Data Control

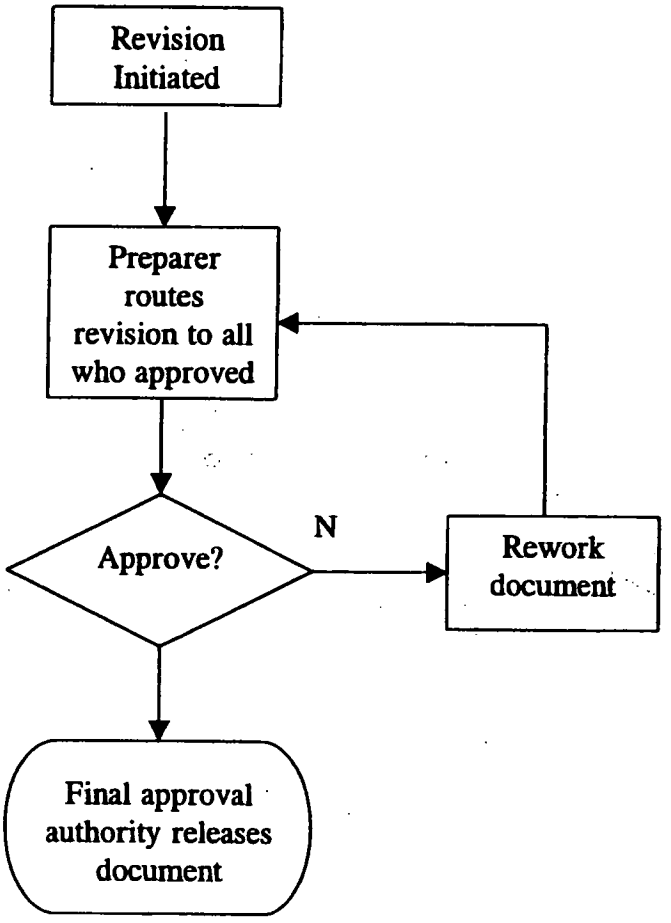
APPENDIX A

New Electronic Document Procedure Flowchart


New Document Created
Document Control Custodian checks document into system and routes for approval
Approved?
N
Originator reworks document
Originator routes document for final approval
Approved?
N
Final approval authority releases document

APPENDIX B

Document Revision Procedure Flowchart


Revision Initiated
Preparer routes revision to all who approved
Approve?
N
Rework document
Final approval authority releases document

NAVSURFWARCENDIVCRANEINST 4855.1A
QMO
15 JUN 1999

NAVSURFWARCENDIVCRANE INSTRUCTION 4855.1A

From: Commander

Subj: QUALITY SYSTEM

Ref: (a) Crane Division Strategic Plan
(b) NAVSURFWARCENDIVCRANEINST 5200.2, Management System
(c) ANSI/ASQC Q9001-1994, Quality Systems - Model for Quality Assurance in Design, Development, Production, Installation, and Servicing

Encl: (1) Crane Division Quality Manual

1. Purpose. To document the Quality System at Crane Division, Naval Surface Warfare Center (NAVSURFWARCENDIV Crane) -- hereinafter referred to as "Crane Division" -- which implements the quality system requirements of references (a) - (c). The Quality System supports the implementation of the strategic plan which encompasses Crane Division's vision, mission, guiding principles and goals.

2. Cancellation. NAVSURFWARCENDIVINST 4855.1, NAVWPNSUPPCENINST 4855.9, and NAVWPNSUPPCENINST 4855.11.

3. Scope. This instruction applies to Crane Division.

4. Policy. Crane Division will have a Quality System in place that complies with the requirements published in the current version of the American National Standards Institute (ANSI)/ American Society for Quality (ASQ) Q9001 standard and associated documentation.

5. Action

a. Directorate Directors shall implement an effective quality system based on the applicable requirements defined in enclosure (1).

CRANE DIVISION QUALITY MANUAL

RECORD OF CHANGES

Revision	Date	Description
A		Incorporate requirements of ISO 9001.

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CHAPTER 1

INTRODUCTION

1.1 PURPOSE. This manual documents the Quality System at the Crane Division, Naval Surface Warfare Center (NAVSURFWARCENDIV Crane) -- hereinafter referred to as "Crane Division" -- and, states policy and objectives, assigns responsibilities, and defines requirements in its implementation. Crane Division's Quality System conforms to the requirements defined in ANSI/ASQC Q9001-1994, Quality Systems - Model for Quality Assurance in Design, Development, Production, Installation, and Servicing (commonly referred to as ISO 9001). The manual, also, serves to instruct and guide employees whose actions affect process, product and service quality, and to inform our customers what controls are implemented to assure product/service quality.

1.2 REFERENCES/APPLICABLE DOCUMENTS

1.2.1 References

a. ANSI/ASQC Q9001-1994 Quality Systems - Model for Quality Assurance in Design, Development, Production, Installation and Servicing

b. ISO 8402:1994 Quality Management and Quality Assurance-Vocabulary

1.2.2 **Applicable Documents.** Documents applicable to the implementation of the Quality System are identified in Appendix A.

1.3 DEFINITIONS. Definitions applicable to this instruction are defined below and in ISO 8402.

a. **Customer.** The recipient of a product/service from any performing organization within Crane Division. Thus, customers may be either internal or external.

b. **Performing Organization.** The organizational unit that is tasked to accomplish work.

c. **Product/Service.** A generic term used to describe the totality of outputs from each process performed whether it is the result of work performed by an individual, an interim process/step or the hardware, computer software, documentation, data and services provided to customers.

d. **Project.** A set of specific work requirements and tasks that must be accomplished within a definite time period. The tasks and project performance can be planned, scheduled, tracked and reported as a coherent work effort.

1.4 DISCUSSION

a. Crane Division performs a wide variety of electronic and ordnance tasks for a large number of customers. As part of Crane Division's engineering, acquisition, logistics and Fleet support responsibilities, Crane Division focuses on improving performance, reducing total ownership cost and improving Fleet support in the following areas: Electronic Warfare; Small Arms; Microelectronics Technology; Conventional Ammunition Engineering; Electronic Module Test & Repair; Pyrotechnics; Microwave Components; Electrochemical Power Systems; Acoustic Sensors Test; Radar Engineering & Industrial Support; Physical Security; Night Vision/Electro-Optics; and Gun Weapon Systems. Direct work within Crane Division is distributed among directorates that are organized around various technologies and services. The directorates are defined in NAVSURFWARCENDIVCRANEINST 5450.1, Naval Surface Warfare Center, Crane Division Organization Manual. To maintain a focus on our customers, the work is organized into projects headed by a project manager or Business Unit Manager.

b. To accommodate the variety of customer requirements for quality, the Quality System consist of the basic quality requirements defined at the corporate level in this Quality Manual and related Crane Division instructions; and supplemented by customer unique and/or product/service/process specific requirements documented at the project/performing organization level.

c. Chapter 2 of this manual corresponds to the quality system requirements of ISO 9001 and incorporates the general concepts of project and process management. Project management deals with interfacing with the customer to identify, understand and document their requirements, and ensuring that tasks are performed within cost and schedule. Process management deals with providing adequate resources required to provide products/services, and ensuring that products/services meet the customer's requirements.

d. This manual is available on Crane Division's Intranet at http*://intranet.crane.navy.mil/qmo/PMODOC.asp*.

1.5 PRECEPTS. This Quality System is based on the following precepts:

a. Responsibility for quality is an integral part of everyone's job.

b. Effective quality efforts are prevention-oriented and process-driven.

c. Crane Division is here to serve the customer.

1.6 QUALITY OBJECTIVES

a. Continually improve the level of customer satisfaction.

b. Continually improve work processes and the quality system.

c. Achieve and maintain a quality system based on ISO 9001 quality system requirements.

CHAPTER 2

QUALITY SYSTEM

2.1 MANAGEMENT RESPONSIBILITY

2.1.1 **Quality Policy.** The quality policy, stated below, is disseminated throughout Crane Division via Quality System briefings, publications, and the Intranet. Quality evaluations of projects/performing organizations ensure that this policy is understood, implemented and maintained.

> ***We will provide products/services that meet our customers' requirements and expectations. We will achieve this through the use of proven processes and personnel, and continual process improvements.***

W. E. SHOTTS

2.1.2 **Organization.** Appendix B shows Crane Division's organizational relationships.

2.1.2.1 Responsibility and Authority

a. ***Senior Management***

(1) The Commander is the ultimate authority and provides vision and direction in the execution and use of Crane Division resources to provide our customers with the products/services that meet their requirements.

(2) The Executive Director and Directorate Directors have the ultimate responsibility to implement quality system policy and directives as directed by the Commander.

b. ***Project Managers*** are appointed by Command/Executive Director/Directorate Directors/Department Directors and serve as the primary contact with external customers. They are responsible and accountable for identifying customer requirements, assessing customer satisfaction, ensuring that products/services meet customer requirements through the use of appropriate performance measures, and ensuring that work is performed on schedule and within cost.

c. ***Performing Organization Managers*** (Department directors, Branch managers and lower level supervisors) serve as leaders/coaches of natural work groups. They are responsible and

accountable for the satisfactory completion of work assigned to their organization. They prioritize resources among work assignments; help employees understand and accomplish their assigned work; and, develop employees and assure they are properly trained. Performing organization managers lead process validation efforts, and work with employees and other managers to find and implement improvements to work processes. As assigned by the Directorate director, performing organization managers serve as Business Unit managers.

d. ***Employees*** are responsible for the quality of the work they perform and for their part in the overall process used to provide products/services to the customers. Employees, individually and/or through the organizational structure, help to develop measures of process effectiveness, collect and analyze work process data, document in-process non-conformances, propose process improvements, evaluate and critique improvement plans, and work with internal suppliers and customers to make the entire work process the best it can be. Employees have the freedom to identify and report any problems related to the product/service, process and Quality System.

e. The ***Crane Division Quality Focal Point*** (QFP) (Quality Management Office (QMO)): 1) serves as Crane Division's primary point of contact (management representative) with internal and external parties on quality system issues; 2) assist in identifying and removing barriers to the full implementation of the quality system; 3) provides advice and guidance to Crane Division's senior management in the implementation of the quality system; and, 4) leads the operation of the Division Quality Team (see paragraph g below).

f. ***Directorate Quality Focal Point*** (QFP) is an individual appointed by the Directorate director and empowered to act on the behalf of the directorate regarding quality system issues. Directorate QFPs: 1) serve as the single point of contact on quality system matters for their directorate; 2) serve as their directorate's representative on the Division Quality Team; 3) communicate quality policy matters within their directorate; 4) coordinate and assist directorate organizations in responding to requests for corrective action and verifies its implementation; 5) coordinate the development and maintenance of directorate level quality system implementing instructions; 6) coordinate the response to incoming Product Quality Deficiency Reports within the directorate; and, 7) assist QMO in the scheduling of internal quality evaluations, coordinating evaluations within the directorate and monitoring applicable corrective actions. Additionally, as requested/directed by their directorate, the QFP assists managers and supervisors in the development and execution of quality system elements and policy, and advises project managers in the preparation of project/quality plans, as applicable. Minimum qualifications for

the Directorate QFP are general knowledge of quality systems as may be gained through experience or in reading ISO 9001 documentation. The list of current Directorate QFP's is maintained on Crane Division's Intranet at ***http://intranet.crane.navy.mil/qmo/default.asp***.

g. ***Crane Division Quality Team*** is a working group chaired by the Crane Division QFP and is comprised of Directorate QFPs and other personnel as designated by the Crane Division QFP. The team participates in the development, evaluation and improvement of the Quality System and performs other actions as assigned by Command or the Crane Division QFP. The Crane Division Quality Team serves in an advisory capacity to the Crane Division QFP in the development of corporate level quality policy and objectives.

h. ***Quality Management Office*** (QMO), through the Strategic Planning Office, is staffed to Command and is responsible for Quality System policy, awareness, assessment and continual improvement. QMO develops and maintains Crane Division's Quality System policies and instructions; and publicizes and provides guidance on the application of Quality System policies, tools and techniques. QMO assesses and reviews quality functions and the degree of compliance with customer imposed requirements, and other directives applicable to products/services and processes. QMO promotes and supports quality improvement efforts and the development of corporate level quality and performance indicators.

i. ***Other Assignments*** are documented by management, as necessary, for the satisfactory accomplishment of quality related functions.

2.1.2.2 Resources. Management is responsible for identifying and providing sufficient resources, including trained personnel, for management, work performance, verification and quality evaluations.

2.1.2.3 Management Representative. The Crane Division QFP is designated as the management representative for Crane Division. As the management representative, the Division QFP is responsible for: 1) ensuring that a Quality System is established, implemented and maintained in accordance with ISO 9001; and, 2) reporting on the performance of the Quality System to senior management for the purpose of review and improvement.

2.1.3 **Management Review.** At least annually, the management representative briefs senior management on the status of the Quality System. The briefing, as a minimum, addresses the Quality System's continuing suitability and its effectiveness in satisfying quality requirements, policy and objectives. A copy of the briefing along with meeting minutes is maintained by QMO.

2.2 QUALITY SYSTEM

2.2.1 **Quality Manual.** This instruction serves as Crane Division's quality manual. It describes the elements of the Quality System and the project/process management approach for ensuring that Crane Division's products/services meet customer requirements and expectations. The manual documents overall quality system policy and requirements; and references Crane Division's quality system implementation procedures. The documentation hierarchy is defined as follows:

a. Level 1 - Quality Manual. The quality manual states the quality system policies, objectives and responsibilities for Crane Division.

b. Level 2 - Instructions & Procedures. Crane Division instructions and performing organization procedures document the principles and operating procedures for one or more quality system elements.

c. Level 3 - Work Instructions. Work instructions are developed by performing organizations to provide detailed step-by-step or general instructions stating how to perform specific processes.

d. Level 4 - Records. Records of quality, including documents such as reports, data sheets, letters and forms that provide objective evidence that quality requirements have been met, are documented and retained according to established procedures.

2.2.2 **Quality System Procedures.** The quality system documentation is comprised of this manual, Crane Division instructions and lower level procedures, as appropriate, that implement the requirements contained herein.

a. Quality system implementing procedures applicable to all directorates are documented in the Quality Manual and Crane Division instructions. These procedures identify a consistent and uniform approach for implementing the quality system requirement across Crane Division. QMO is responsible for the development and maintenance of this manual and accompanying Crane Division instructions. Crane Division instructions are prepared and authorized per NAVSURFWARCENDIVCRANEINST 5215.1, Directives System. Appendix A provides a listing of applicable Division level instructions. A master list of Crane Division Quality System instructions is maintained on the Crane Division's Intranet at ***http://intranet.crane.navy.mil/qmo/PMODOC.asp***.

b. Quality system procedures applicable to only one organization (e.g., directorate, department) are documented at the appropriate level. These instructions provide detailed

procedures for implementing quality requirements that are uniquely applicable to that organization's processes or customers. These procedures are written to comply with applicable ISO 9001 requirements.

c. Performing organizations shall effectively implement the quality system and its documented procedures.

2.2.3 **Quality Planning.** This manual serves as the quality plan for Crane Division. Project/performing organization managers develop and approve, as necessary, plans at their level to define and document how project, process and quality requirements, derived from work authorization documents, are satisfied. Such plans are tailored to the project/performing organization and identify the activities, as appropriate, to meet the specific requirements for projects, products/services and customer requirements including:

a. The identification and acquisition of any controls, processes, inspection equipment, fixtures, resources and skills that may be needed to achieve the required quality.

b. Ensuring the compatibility of the design, the production process, installation, servicing, inspection and test procedures, and the applicable documentation.

c. The updating, as necessary, of quality control inspection and testing techniques, including the development of new instrumentation.

d. The identification of any measurement requirement involving capability that exceeds the known state of the art in sufficient time for the needed capability to be developed.

e. The identification of suitable verification at appropriate stages in the realization of the product/service.

f. The clarification of standards of acceptability for all features and requirements, including those which contain a subjective element.

g. The identification and preparation of quality records. (See Appendix C.)

Managers are responsible for ensuring that the plans are reviewed and updated, if appropriate, at least annually. Copies of these plans are provided, as needed, to appropriate external and internal customers.

2.3 CONTRACT (WORK AUTHORIZATION) REVIEW. Project/business unit managers are responsible for reviewing work authorization documents and their amendments to ensure that: requirements are

adequately defined and documented; any differences from cost estimates are resolved; capability exist to meet the work authorization document requirements; and terms of the work authorization document are acceptable. Projects/performing organizations are responsible for ensuring that amendments to work authorization documents are communicated to those functions affected by the amendment. Records of these reviews shall be maintained. See NAVSURFWARCENDIVCRANEINST 4855.3.

2.4 DESIGN AND DEVELOPMENT CONTROL. Projects/performing organizations assigned design and development tasks are responsible for preparing documented procedures to control and verify the design of products to ensure that specific requirements are designed into the product/service and that other pertinent issues (including environmental concerns) are considered at the conceptual and design stages of the product/service. NAVSURFWARCENDIVCRANEINST 4855.2 provides guidance applicable to the control and verification of the design of a product/service including:

a. Preparation of design and development plans that describe or reference the controls and responsibilities related to the design/development activity including the assignment of qualified personnel and adequate resources. Updating the plan as the design evolves.

b. Defining the organizational and technical interfaces between the different groups which input to the design process, and documenting, transmitting, and regularly reviewing the necessary information.

c. Identifying, documenting, and reviewing the selection of design input requirements relating to the product/service including applicable statutory and regulatory requirements. Resolving incomplete, ambiguous, or conflicting requirements with those responsible for imposing these requirements. Taking into consideration the results of any work authorization review activities.

d. Documenting and expressing design output in terms that can be verified and validated against design input requirements. Reviewing design output documents prior to release to ensure that the design output: meets the design input requirements; contains or makes reference to acceptance criteria; and identifies those characteristics of the design that are crucial to safe and proper functioning/use of the product/service (e.g., operating, storage, handling, maintenance and disposal requirements).

e. Planning and conducting formal documented reviews of the design at appropriate stages. Ensuring that participants at the design reviews include representatives of all functions

concerned with the design stage being reviewed, as well as other specialist personnel. Maintaining records of design reviews.

f. Performing design verifications at appropriate stages of the design to ensure that the design stage output meets the design stage input requirements. Recording design verification measures.

g. Performing design validation to ensure that the product/service conforms to defined user needs and/or requirements.

h. Identifying, documenting, reviewing and approving by authorized personnel all design changes and modifications prior to their implementation.

2.5 DOCUMENTATION AND DATA CONTROL. Projects/performing organizations are responsible for assuring that documents and data affecting the quality of their products/services and processes are controlled and managed, including distribution and changes, to maintain process integrity. Documents and data shall be reviewed and approved for adequacy by authorized personnel prior to issue. A master list or equivalent document control procedure shall be established and readily available to identify the current revision status of documents and preclude the use of invalid and/or obsolete documents. These controls shall ensure that: (1) the pertinent issues of appropriate documents are available at all locations where operations essential to the effective functioning of the quality system are performed; (2) invalid and/or obsolete documents are promptly removed from all points of issue or use, or otherwise assured against unintended use; and (3) any obsolete documents retained for legal and/or knowledge preservation purposes are suitably identified. NAVSURFWARCENDIVCRANEINST 4855.8 provides applicable requirements for the development, control and issuance of documents including the control of documents of external origin.

2.6 PURCHASING. The Supply Directorate coordinates purchasing of products/services. Procurements are developed and administered in accordance with applicable Federal Acquisition Regulations (FAR) and Defense Federal Acquisition Regulations Supplement (DFARS) as documented for general procurements in the Acquisition Request Preparation Guide (available on Crane Division's Intranet at ***http://intranet.crane.navy.mil/Library/CranesDocuments/arpguide.pdf***) and for credit card buys in the Government-Wide Commercial Purchase Card Program Manual. Where appropriate and as authorized by the FAR/DFAR, the above documents are supplemented by performing organization procedures to ensure that:

a. Suppliers of products/services are evaluated and selected based on their ability to meet contract requirements

including quality system and any specific quality assurance requirements; and records of acceptable contractors are established and maintained. The type and extent of controls over a contractor are defined and dependent upon the type of product/service, the impact of the product/service on the quality of the final product/service and, where applicable, on quality audit reports and/or quality records of previously demonstrated capability and performance of the contractor.

b. Procurement documents shall contain data that clearly describes the product/service ordered, including where applicable:

(1) The type, class, grade or other precise identification.

(2) The title or other positive identification, applicable issues of specifications, drawings, process requirements, inspection instructions and other relevant technical data including requirements for approval or qualification of product/service, procedures, process equipment and personnel.

(3) The title, number and issue of the quality system standard to be applied. Procurement documents shall be reviewed and approved for adequacy of the specified requirements prior to release.

c. Where it is proposed to verify the procured product/service at the contractor's premises, verification arrangements and the method of product release are to be included in the purchasing document.

d. Where specified in work authorization documents, the customer or their representative shall be afforded the right to verify at the contractor's premises and the performing organization's premises that the procured product/service conforms to specified requirements. This verification shall not be used by the performing organization as evidence of effective control of quality by the contractor. The verification by the customer does not absolve the performing organization of its responsibility to provide acceptable product/service, nor does it preclude subsequent rejection by the customer.

2.7 CONTROL OF CUSTOMER SUPPLIED/OWNED MATERIAL. Unless specified otherwise by the customer, customer-supplied material, from time of receipt until the time it is consumed or transferred out of Crane Division, is verified, stored and maintained in accordance with the requirements and procedures defined by the project/performing organization. Items that are subsequently lost, damaged or otherwise deemed unsuitable for use are recorded

and reported to the customer through applicable non-conforming product or corrective action process.

2.8 PRODUCT IDENTIFICATION AND TRACEABILITY. Performing organizations are responsible for documenting procedures for the identification of product/material from receipt and throughout production, testing, delivery and installation, as applicable. Where traceability is a requirement, the performing organization establishes and maintains a documented process for the unique identification of individual product/material or batches. Records of this identification are maintained by the performing organization, as defined by their local procedures.

2.9 PROCESS CONTROL. Crane Division employs a process management approach to ensure that all processes - whether design and development, test and evaluation, acquisition, manufacturing and repair, logistics support, in-service support, or administrative support - are planned and developed to ensure that they are carried out under controlled conditions to meet customer requirements and expectations. Performing organizations ensure that process outputs are clearly defined and processes are designed to satisfy customer requirements. Key processes, as identified by the performing organization, are validated to prove process capability. Process validation is accomplished through demonstrations using operational process personnel, documented procedures, facilities, equipment and material. Validation includes, as appropriate, the qualification of personnel, facilities, equipment and documentation for the process. Processes are revalidated: when significant changes, as defined by the performing organization, occur in the process; based on the results of customer complaints; and when process data indicates a degradation in process effectivity or efficiency. Validation records are maintained by the performing organization unless otherwise stipulated by the customer. This approach includes the use of controlled conditions:

a. Documented procedures and criteria for workmanship.

b. Knowledgeable and qualified personnel, as applicable.

c. Suitable equipment and facilities.

d. Compliance with reference standards, codes, safety requirements, quality plans or other applicable documented procedures.

e. Process measures, as appropriate.

2.10 INSPECTION AND TESTING. Performing organizations are responsible for documenting the inspection and test processes used to ensure the quality of material throughout the processing

stages. The use of statistical methods, including sampling, is encouraged where appropriate.

2.10.1 **Receiving Inspection and Testing.** Performing organizations shall ensure that incoming products are not used until they have been inspected or otherwise verified as conforming to specified requirements. Verification of conformance to specified requirements shall be in accordance with the quality plan and/or documented procedures. In determining the amount and nature of receiving inspection, consideration shall be given to the amount of control exercised at the contractor's premises and the recorded evidence of conformance provided. Where incoming product is released for urgent production purposes prior to verification, the product shall be positively identified and recorded in order to permit immediate recall and replacement in the event of nonconformity to specified requirements.

2.10.2 **In-Process Inspection and Testing.** Performing organizations shall inspect and test the product as required by the quality plan and/or documented procedures. Products shall be held until the required inspection and tests have been completed or the necessary reports have been received and verified.

2.10.3 **Final Inspection and Testing.** Performing organizations shall carry out all final inspection and testing in accordance with the quality plan and/or documented procedures to complete the evidence of conformance of the finished product to the specified requirements. The quality plan and/or documented procedures for final inspection and testing shall require that all specified inspections and tests, including those specified either on receipt of product or in-process, have been carried out and that the results meet the specified requirements. No product shall be dispatched until all activities specified in the quality plan and/or documented procedures have been satisfactorily completed and the associated data and documentation are available and authorized.

2.10.4 **Inspection and Test Records.** Records of inspections and tests shall be established and maintained by the performing organization as evidence that the product has been inspected and/or tested. These records shall clearly show whether the product has passed or failed the inspections and/or tests according to defined acceptance criteria. Where the product fails to pass any inspection and/or tests, the procedures for control of nonconforming product shall apply. Records shall identify the inspection authority responsible for the release of product.

2.11 CONTROL OF INSPECTION, MEASURING AND TEST EQUIPMENT

2.11.1 **General.** Procedures and responsibilities for the control, calibration and maintenance of inspection, measuring and test equipment (including test software) used to demonstrate the conformance of product to specified requirements are established and documented in NAVSURFWARCENDIVCRANEINST 4855.20. The Calibration Laboratory manages, operates and maintains Crane Division's calibration program. Performing organizations are responsible for ensuring that calibration status of equipment is maintained as applicable. Where test software or comparative references such as test hardware are used as suitable forms of inspection, they shall be checked to prove they are capable of verifying the acceptability of product, prior to release for use during production, installation or servicing; and, shall be rechecked at prescribed intervals. The extent and frequency of such checks shall be established and records maintained as evidence of control. Where the availability of technical data pertaining to the inspection, measuring and test equipment is a specified requirement, such data shall be made available, when required by the customer or customer's representative, for verification that the inspection, measuring and test equipment is functionally adequate.

2.11.2 **Control Procedures.** Control of inspection, measuring and test equipment is achieved by:

a. Determining the measurements to be made and the accuracy required, and selecting the appropriate inspection, measuring and test equipment that is capable of the accuracy and precision necessary.

b. Identifying all inspection, measuring and test equipment that can affect product quality.

c. Calibrating and adjusting the inspection, measuring and test equipment at prescribed intervals, or prior to use, against certified equipment having a known valid relationship to internationally or nationally recognized standards. Where no such standards exist, the basis used for calibration will be documented.

d. Defining the process employed for the calibration of inspection, measuring and test equipment including details of equipment type, unique identification, location, frequency of checks, check method, acceptance criteria and the action to be taken when results are unsatisfactory.

e. Identifying inspection, measuring and test equipment with suitable indicator or approved identification label/tag to show the calibration status.

f. Maintaining calibration records for inspection, measuring and test equipment.

g. Assessing and documenting the validity of previous inspection and test results when inspection, measuring and test equipment is found to be out of calibration.

h. Ensuring that the environmental conditions are suitable for the calibration, inspections, measurements and tests being carried out.

i. Ensuring that the handling, preservation and storage of inspection, measuring and test equipment is such that the accuracy and fitness for use is maintained.

2.12 INSPECTION AND TEST STATUS. Performing organizations are responsible for documenting the inspection and test status of products. As a minimum, the inspection and test status of items in-process is identified by means of a traveler (a document or record that accompanies and documents the status a product during the processing cycle). Completed travelers are maintained by the performing organization as documented. Products placed in the stock system or shipped to the end user are identified, as appropriate, as to their condition status. The condition status of serviceable items is authenticated with an inspection indication device or by other documented means. See NAVSURFWARCENDIVCRANEINST 4855.6.

2.13 CONTROL OF NONCONFORMING PRODUCT. Performing organizations are responsible for documenting procedures for the identification, documentation, evaluation, segregation and disposition of nonconforming product. The procedure or the applicable project/quality plan identifies the authority for approving waivers, deviations and engineering change proposals. In general, these procedures include the marking/tagging of products that fail inspection as reject and segregating the nonconforming product pending disposition. As appropriate, Material Review Boards (MRBs) are established by the performing organization for the evaluation and disposition of nonconforming product. Disposition of nonconforming product may include: rework to meet the specified requirements; accept with or without repair by concession; regrade for alternative applications; or, reject or scrap. If required by the work authorization document, the proposed use or repair of nonconforming product shall be reported for concession to the customer or customer's representative. The description of the nonconformity that has been accepted and of repairs shall be recorded to denote the actual condition. Repaired and/or reworked product shall be reinspected in accordance with the quality plan and/or documented procedures.

2.14 CORRECTIVE AND PREVENTIVE ACTION. Performing organizations are responsible for establishing and maintaining documented procedures for implementing corrective and preventive actions. Any action taken to eliminate the causes of actual or potential nonconformities shall be to the degree appropriate to the magnitude of the problems and commensurate with the risks encountered. The performing organizations shall implement and record any changes to the documented procedures resulting from corrective and preventive action.

2.14.1 **Corrective Action.** Performing organizations are responsible for establishing and maintaining internal corrective action procedures for the effective handling of customer concerns/complaints, product nonconformances, and in-process discrepancies. These procedures shall, also, identify the methods used to investigate the cause of the complaint/nonconformance/discrepancy, the steps for determining corrective action to eliminate the cause of the complaint/nonconformance/discrepancy, and the follow-up activity to ensure corrective action is taken and is effective. NAVSURFWARCENDIVCRANEINST 4855.21 addresses the processing of incoming Product Quality Deficiency Reports. NAVSURFWARCENDIVCRANEINST 4855.22 defines the responsibilities and requirements for processing of Trouble and Failure Reports related to tasks performed for Strategic Systems Programs.

2.14.2 **Preventive Action.** Performing organizations are responsible for establishing and maintaining procedures for preventive actions. These procedures shall identify the use of appropriate sources of information to detect, analyze and eliminate potential causes of nonconformances; the determination of the steps needed to deal with any problems requiring preventive action; the initiation of preventive action and application of controls to ensure that the action is effective; and, the means to ensure that relevant information on actions taken is submitted for management review.

2.15 HANDLING, STORAGE, PACKAGING, PRESERVATION AND DELIVERY

2.15.1 **General.** Performing organizations are responsible for documenting and maintaining procedures for the handling, storage, packaging, preservation, and delivery of products within their scope of operations.

2.15.2 **Handling.** Handling methods to prevent damage and deterioration are implemented as required. Electrostatic Discharge (ESD) handling and precautions are documented in NAVSURFWARCENDIVCRANEINST 4855.10. NAVSURFWARCENDIVINST 4855.19 provides requirements for controlling certain materials from exposure to mercury.

2.15.3 **Storage.** When required, special provisions are established and implemented. E.g., ESD sensitive items (see NAVSURFWARCENDIVCRANEINST 4855.10), limited life items (see NAVSURFWARCENDIVCRANEINST 4855.4) and items requiring environmental controls.

2.15.4 **Packaging.** Packaging and marking of products is performed in accordance with customer requirements and applicable military/commercial standards/practices.

2.15.5 **Preservation.** Appropriate methods or best commercial practices as identified by customer requirements or appropriate military/commercial standards for the preservation and segregation are employed for the performing organization's products.

2.15.6 **Delivery.** Appropriate controls are employed to protect the quality of products following final inspection and acceptance. Where applicable, these controls extend to include delivery to destination.

2.16 CONTROL OF QUALITY RECORDS. Objective evidence of performance is established and maintained by the performing organization to demonstrate quality achievement, to assess quality improvement progress, for analysis of trends, and to identify the need for and effectiveness of corrective and preventive actions. Performing organizations shall establish and maintain documented procedures for the identification, collection, indexing, access, filing, storage, maintenance and disposition of quality records (see NAVSURFWARCENDIVCRANEINST 5216.1, Correspondence Manual, for general guidance). Pertinent quality records from contractors shall also be an element of this data. Quality records shall be legible. The records shall be stored and retained in such a way that they are readily retrievable in facilities that provide a suitable environment to prevent damage or deterioration and to prevent loss. Retention times of quality records shall be established and recorded. If required by the work authorization document, quality records shall be made available for evaluation by the customer or customer's representative for an agreed period. Appendix C provides a listing of quality records required by ISO 9001, responsibility for their maintenance and minimum retention periods.

2.17 INTERNAL QUALITY EVALUATIONS. Internal quality evaluations, including both Quality System Evaluations and directorate level evaluations, are conducted to assess the implementation and effectiveness of the Quality System, to assess compliance of operations to governing requirements, and to identify opportunities for improvement. Procedures for the planning, performance, documentation, reporting and follow-up of quality evaluations are documented in

NAVSURFWARCENDIVCRANEINST 4855.12. QMO is responsible for coordinating corporate level quality evaluations and schedules these evaluations based on the status and importance of the activity to be audited and customer requirements. Personnel assigned to perform the evaluations are independent of the activities being evaluated. The results of the evaluation are recorded and brought to the attention of personnel having responsibility for the activity evaluated. Management personnel are responsible for seeing that timely corrective action is taken on the deficiencies identified during the evaluation. Follow-up activities are conducted to verify and record the implementation and effectiveness of corrective action taken. Performing organizations conduct internal evaluations to identify opportunities for improvement.

2.18 TRAINING. Skill requirements for personnel performing services that directly affect the quality of products are documented by the performing organization either by individual or job title/classification. For specific tasks requiring specialized skills, personnel are qualified on the basis of appropriate education, training and/or experience, as required. Training records are maintained by the performing organization.

2.19 SERVICING. Not applicable to this scope.

2.20 STATISTICAL TECHNIQUES

2.20.1 **Identification of Need.** When applicable, performing organizations identify in their quality plan the statistical techniques required for establishing, controlling and verifying process capabilities and product characteristics.

2.20.2 **Procedures.** Where statistical techniques are required, performing organizations establish and maintain documented procedures to implement and control the application of the statistical techniques.

APPENDIX A

APPLICABLE QUALITY SYSTEM DOCUMENTS

PARAGRAPH	QUALITY SYSTEM DOCUMENTS
2.1 Management Responsibility	Documented in this instruction.
2.2 Quality System	Documented in this instruction.
2.3 Work Authorization Review	4855.3
2.4 Design and Development Control	4855.2
2.5 Documentation and Data Control	4855.8
2.6 Purchasing	Documented in Supply Directorate Acquisition Request Preparation Guide and Government-Wide Commercial Purchase Card Program Manual.
2.7 Control of Customer Supplied/Owned Material	See Note 1.
2.8 Product Identification and Traceability	See Note 1.
2.9 Process Control	4855.18
2.10 Inspection and Testing	See Note 1.
2.11 Control of Inspection, Measuring and Test Equipment	4855.20
2.12 Inspection and Test Status	4855.6
2.13 Control of Nonconforming Product	See Note 1.
2.14 Corrective and Preventive Action	4855.12, 4855.21, 4855.22
2.15 Handling, Storage, Packaging, Preservation and Delivery	4855.4, 4855.10, 4855.19
2.16 Control of Quality Records	Documented in this instruction.
2.17 Internal Quality Evaluations	4855.12
2.18 Training	See Note 1.
2.19 Servicing	Not applicable to this scope.
2.20 Statistical Techniques	See Note 1.

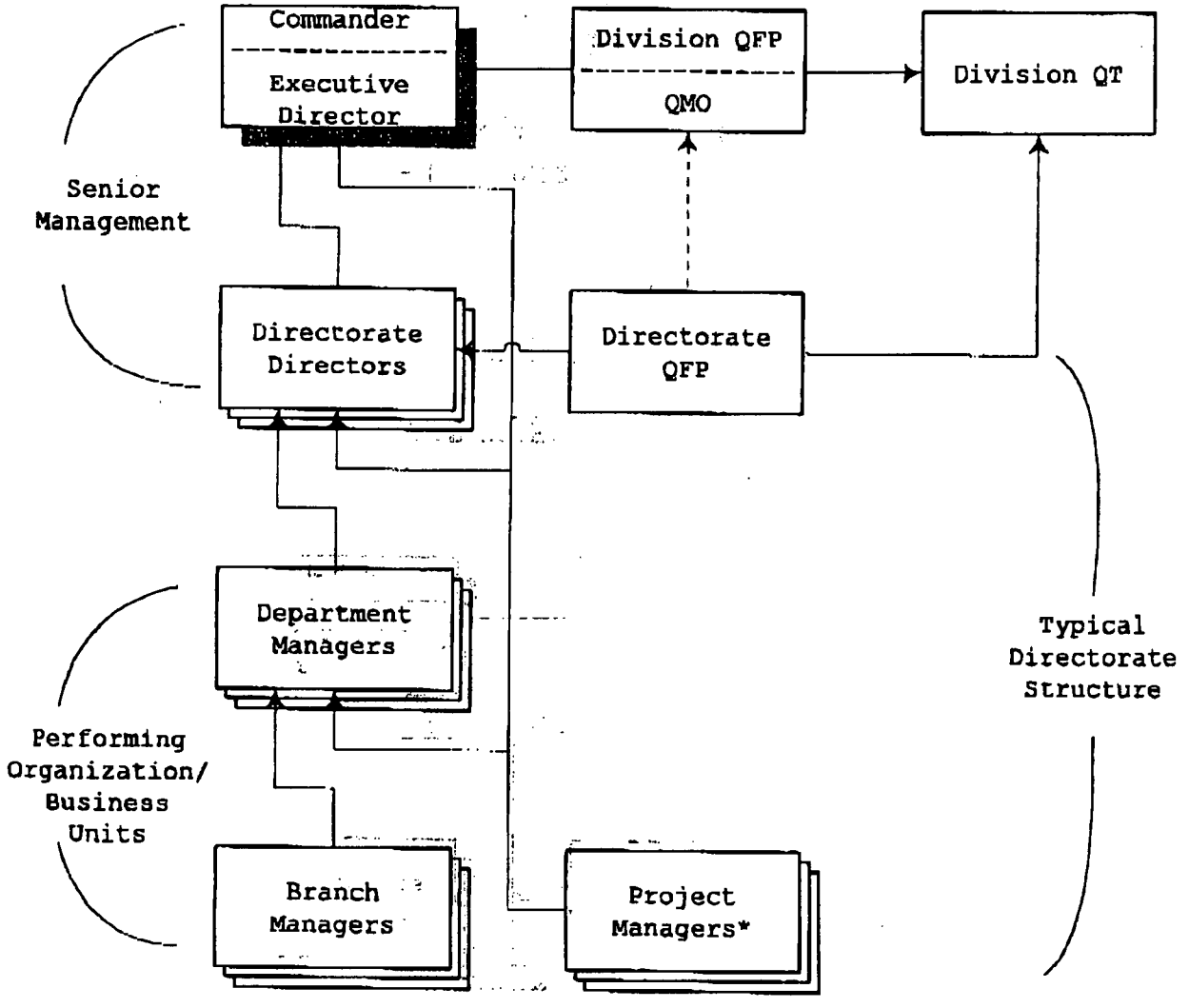
Notes: 1. To be developed, as applicable, by the performing organization in compliance with ISO 9001 requirements.

QUALITY SYSTEM DOCUMENTS

NAVSURFWARCENDIVCRANEINST 4855.2	Design and Development Quality Requirements and Guidelines
NAVSURFWARCENDIVCRANEINST 4855.3	Work Authorization Document Review
NAVSURFWARCENDIVCRANEINST 4855.4	Shelf Life Management
NAVSURFWARCENDIVCRANEINST 4855.6	Inspection Indication Devices and Material Identification
NAVSURFWARCENDIVCRANEINST 4855.8	Technical Documentation Development, Control and Issuance
NAVSURFWARCENDIVCRANEINST 4855.10	Electrostatic Discharge (ESD) Control Program
NAVSURFWARCENDIVCRANEINST 4855.12	Quality Evaluation Program
NAVSURFWARCENDIVCRANEINST 4855.18	Soldering Program
NAVSURFWARCENDIVINST 4855.19	Identification and Control Systems for Mercury Free, MIC Level I (Non-Nuclear) and Subsafe Materials
NAVSURFWARCENDIVCRANEINST 4855.20	Calibration Program
NAVSURFWARCENDIVINST 4855.21	Incoming Product Quality Deficiency Reports
NAVSURFWARCENDIVCRANEINST 4855.22	Strategic Weapon System Trouble and Failure Report Program

APPENDIX B

ORGANIZATION CHART



* Project Managers may be appointed at any of the indicated organizational levels.

APPENDIX C

LIST OF QUALITY RECORDS

Paragraph Number	Type of Record	Maintained By	Minimum Retention Period
2.1.3	Management Review Minutes/Briefings	Quality Management Office	3 years
2.3.3	Funding Document Acceptance/ Non-Acceptance Form	Funds Administrators	7 years
2.4	Design Review/Verification Records	Performing Organization	TBD*
2.6	Acceptable Subcontractors	Performing Organization	TBD
2.7	Records of Lost/Stolen Customer-Supplied Product	Performing Organizations	TBD
2.8	Identification and Traceability Records	Performing Organization	TBD
2.9	Certifications of Personnel, Equipment & Processes	Performing Organization	TBD
2.10	Inspection and Test Records	Performing Organization	TBD
2.11	Inspection, Measuring and Test Equipment Calibration Records	Calibration Laboratory/ Equipment Custodians	TBD
2.13	Records of Nonconforming Material	Performing Organization	TBD
2.14.1	Corrective Action Records	Performing Organization	TBD
2.14.1	Product Quality Deficiency Reports	Fleet Liaison Office	TBD
2.14.1	SSP Trouble Failure Reports	SSP related Performing Organizations	TBD

Paragraph Number	Type of Record	Maintained By	Minimum Retention Period
2.17	Evaluation Schedule	Quality Management Office	2 years
2.17	Records of Internal and External Quality System Evaluation Results and Follow-up Actions	Quality Management Office	2 years
2.18	Training Records	Immediate Supervisor	TBD

*TBD - To Be Determined by the responsible organization.

Note: These are the minimum records to be maintained as identified by ISO 9001.

DEPARTMENT OF THE NAVY
CRANE DIVISION
NAVAL SURFACE WARFARE CENTER
300 HIGHWAY 361
CRANE, INDIANA 47522-5000

IN REPLY REFER TO:
NAVSURFWARCENDIVINST 5090.2B
095

10 SEP 1999

NAVSURFWARCENDIV INSTRUCTION 5090.2B

From: Commander

Subj: HAZARDOUS WASTE MANAGEMENT PLAN

Ref:
(a) OPNAVINST 5090.1B, CH-1, Environmental and Natural Resources Program Manual (NOTAL)
(b) OPNAVINST 4110.2, Hazardous Material Control and Management (NOTAL)
(c) 40 CFR 261-270
(d) 40 CFR 279, Standards for the Management of used Oil
(e) NAVSURFWARCENDIVINST 5090.6 Hazardous Materials Control and Management Program
(f) NAVSEA OP 5 Volume 1 Sixth Revision, Ammunition and Explosives Ashore Safety Regulations for Handling, Storing, Production, Renovation and Shipping.
(g) NAVSURFWARCENDIVINST 5090.5A, Oil and Hazardous Substances Spill Contingency Plan.
(h) AR 200-1, Environmental Protection and Enhancement.
(i) 49 CFR 171-179, Department of Transportation Hazardous Material Regulations.
(j) 29 CFR 1910.120
(k) NSWC Crane Division Pollution Prevention Plan, June 1999

Encl:
(1) Hazardous Waste Management Plan Definitions and Acronyms
(2) Hazardous Waste Management Plan for NAVSURFWARCENDIV Crane

1. Purpose. To state the Crane Division, Naval Surface Warfare Center (NAVSURFWARCENDIV Crane) policy for the management and minimization of hazardous wastes in accordance with references (a) through (k).

2. Cancellation. NAVSURFWARCENDIVINST 5090.2A Hazardous Waste Management Plan, 25 July 1994. This instruction shall be effective ninety (90) days from the date of signature.

3. Definitions. Definitions for the purpose of this instruction are found in enclosure (1).

4. Discussion. NAVSURFWARCENDIV Crane and its Tenant Activities generate a significant amount of hazardous waste annually. This includes solvent wastes such as degreasers, cleaners, and thinners, as well as reactive wastes and by-products from explosive operations. This plan incorporates options available to Crane organizations to minimize hazardous wastes and to eliminate spent solvent wastes in accordance with references (a) through (k). Solvent degreasing tanks and equipment are available through a solvent rental program. This program includes solvent removal and replacement on schedules that are determined by the amount of use. The solvents are much less toxic than many of those frequently used in the past. The spent solvents are recycled. A rag rental program has been made available through a contract administered by the Supply Directorate. This program will provide quality shop towels that are laundered and exchanged to avoid disposal. The regulatory requirements for hazardous waste minimization combined with the increasing costs and limited means of disposal of hazardous wastes has created a need to redirect our actions and operations in order to maintain regulatory compliance. Furthermore, due to the decreasing Department of Defense (DOD) workforce, but continued operational levels at Crane, new policy is needed to ensure adequate protection of natural resources.

5. Scope. This instruction applies to NAVSURFWARCENDIV Crane, all Tenant Activities and Contractors. This instruction does not apply to Crane detachments at Fallbrook or Hawthorne.

6. Policy

a. The guidelines in this instruction will be followed to assure Crane's compliance with regulations set forth in references (a) through (k). Specific policy guidance on minimization, prepared by Public Works Directorate Pollution Prevention Office (PPO), is provided in reference (k).

b. Federal and State regulations govern all aspects of hazardous waste management (handling, containerization, labeling, storage, transportation, treatment, disposal, minimization, etc.) to monitor the wastes from cradle to grave.

Crane's policy directly reflects this concept and is tightly coupled with requirements for minimizing hazardous waste as well as for eliminating the disposal of used solvents. Emphasis is to be put on tightly controlling the procurement of hazardous materials in amounts no greater than will be necessary to complete or sustain programs and projects. The Supply Directorate has made many changes in procurement procedure that greatly enhance their ability to accommodate the hazardous material program. This includes a strong shelf life management program. The Supply Directorate and the PPO are working to determine specific user preferred brands of hazardous materials. This could enable the Supply Directorate to reduce the very large number of active Material Safety Data Sheets (MSDS) that are maintained.

c. The substitution of non-hazardous or less hazardous chemicals in Crane processes must be given highest priority. Hazardous material requisitions for materials that are not on the Authorized Use List (AUL) are screened by PPO. PPO will require that alternative products already on the AUL be evaluated as a substitution. Justification to add new products to the AUL will be required. Special considerations must be given to planning. The procurement of the hazardous materials needed to support operations should be carefully planned to avoid having excess chemicals. Operations will be designed to eliminate or reduce the use of hazardous chemicals. Dry processes will be selected over those that generate volumes of hazardous waste contaminated water or liquid chemical wastes. Recycling and reclamation of spent solvents will be practiced. Minimization methods will be employed in all processes.

7. Action

a. All Directors and Heads of Tenant Activities shall:

(1) Ensure that the policy guidelines prescribed in this instruction are followed.

(2) Advise all personnel involved in any aspect of hazardous waste management of Crane policy requirements as they are promulgated.

(3) Ensure that all personnel involved in any aspect of hazardous waste management attend appropriate hazardous waste management training courses, include applicable hazardous waste assignments in the position descriptions of pertinent personnel, and maintain the names of employees who work with hazardous

waste along with appropriate documentation concerning their training. Copies of this documentation should be forwarded, upon completion of training, to the Environmental Protection Department and kept current when personnel assignments change. This documentation will be inspected annually in October.

b. PPO shall: coordinate the hazardous waste minimization efforts of NAVSURFWARCENDIV Crane as well as authorizing the Hazardous Waste Minimization Plan required by references (a), (b), (e) and (k).

c. The Environmental Protection Department shall: administer the hazardous waste management program for NAVSURFWARCENDIV Crane. This includes updating policy as new State and Federal regulations are promulgated, and enforcing policy guidelines to attain compliance with all pertinent regulations and DOD requirements. Create training regulations for the Environmental Protection Department in accordance with references (a), (c), (d) and (j).

d. The Occupational Safety and Health Department shall: provide training to NAVSURFWARCENDIV Crane employees as required by this directive, and to meet the requirements of references (a), (b) and (f).

8. Reports. The Environmental Protection Department is responsible for the preparation of:

a. Hazardous Waste Annual Report to be submitted to Naval Facilities Engineering Services Center.

b. United States Environmental Protection Agency (USEPA) Hazardous Waste Biennial Report. This report is submitted to IDEM as authorized by the USEPA to administrator this RCRA requirement.

c. Any reporting requirements established per the USEPA/IDEM Hazardous Waste Part B Permit.

d. The Environmental Protection Department will review this instruction annually to ensure it is complete, current and updated as often as necessary to insure regulatory compliance.

e. Other reporting requirements are provided in the Hazardous Waste Management Plan in enclosure (2).

9. Forms. Several forms are used in the execution of NAVSURFWARCENDIV Crane's hazardous waste management program.

a. The following forms are available from the Environmental Protection Department, at Building 3260, extension 1132, (The Environmental Protection Department will assist in obtaining the necessary forms):

(1) Central Storage Facility Log Sheet (NAVSURFWARCENDIVCRANE 6280/5 (Rev. 6-25))

(2) Contaminated Scrap Manifest (NAVSURFWARCENDIVCRANE 6280/4 (Rev. 6/99))

(3) Daily Inspection Log - Open Burning Areas at Ammunition Burning Grounds (NAVSURFWARCENDIVCRANE 5090/7 (Rev. 6/99))

(4) Daily Inspection Log - Sludge Burning Pans (SBP) at Ammunition Burning Grounds (NAVSURFWARCENDIVCRANE 5090/3 (Rev. 6/99))

(5) Environmental Protection Daily Inspection Log - Demolition Range and Open Burning Areas (NAVSURFWARCENDIVCRANE 5090/1 (Rev. 6/99))

(6) Environmental Protection Weekly Inspection Log - Central Storage Facility (NAVSURFWARCENDIVCRANE 5090/4 (Rev. 6/99))

(7) Hazardous Waste Generation Log (NAVSURFWARCENDIVCRANE 6280/2 (Rev. 6-25))

(8) Hazardous Waste Profile Sheet (NAVSURFWARCENDIVCRANE 6280/11 (Rev. 6/99))

(9) Hazardous Waste Program Control Record (NAVSURFWARCENDIVCRANE 6260/6 (Rev. 6/99))

(10) Laboratory Waste Inventory (NAVSURFWARCENDIVCRANE 6280/6 (Rev. 6/99))

(11) Solid and Hazardous Waste Site Inspection Log (NAVSURFWARCENDIVCRANE 5090/2 (Rev. 6/99))

(12) Waste Accumulation Site Weekly Inspection Checklist (NAVSURFWARCENDIVCRANE 6280/1 (Rev. 6-25))

b. Disposal Turn-in Document (DD 1348-1) is available from Building 41 ASRS, Stock No. 0102LF1153800. The most current version is to be used.

c. Ammunition Transfer Record DA 4508 (1 May 76) is available from the Crane Army Ammunition Activity. The most current version is to be used.

T.SCOTT WETTER

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List A1-1, C1
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FOP 1581 Fil

HAZARDOUS WASTE MANAGEMENT PLAN DEFINITIONS AND ACRONYMS

HAZARDOUS WASTE MANAGEMENT PLAN DEFINITIONS

1. At or Near the Point of Generation. The area where wastes initially accumulate which is under the control of the operator of the process generating the waste.

2. Code of Federal Regulations (CFR). The Code of Federal Regulations (CFR) consists of 50 titles representing broad areas subject to Federal regulation. All general and permanent regulations published in the daily Federal Register by executive agencies and departments of the Federal government appear in the CFR, which is updated annually. All regulations issued by the EPA under the subject heading "Protection of the Environment" are codified in Title 40 of the CFR.

3. Combustible Liquid. A liquid having a flash point of 140°F (37.8°C) or higher.

4. Container. Any portable device, in which a material is stored, transported, treated, disposed of, or otherwise handled.

5. Contamination. Degradation as a result of human activities, of natural water, air or soil quality to the extent that its usefulness is impaired.

6. Contingency Plan. A document setting out an organized, planned, and coordinated course of action to be followed in case of a fire, explosion, or release of hazardous waste or hazardous waste constituents that could threaten human health or the environment.

7. Corrosive Material. As defined by DOT, a corrosive material is a liquid or solid that causes visible destruction or irreversible alterations to human skin tissue at the site of contact. In the case of leakage from packaging, a corrosive material is a liquid that has a severe corrosion rate on steel.

8. Corrosive Waste (Corrosivity). As defined by RCRA, an aqueous waste that has a pH </=2.0 or >/=12.5, or has a corrosion rate > 6.35mm (0.250 inch) per year at 55°C.

9. Dike. An embankment or ridge of either natural or man-made materials used to prevent the movement of liquids, sludge, solids, or other materials.

10. Discharge or Hazardous Waste Discharge. The accidental or intentional spilling, leaking, pumping, pouring, emitting,

emptying, or dumping of hazardous waste into or on any land or water.

11. Disposal. The discharge, deposit, injection, dumping, spilling, leaking, or placing of any solid waste or hazardous waste into or on any land or water so that such solid waste or hazardous waste or any constituent thereof may enter the environment or be emitted into the air or discharged into any waters, including ground waters.

12. Disposal Facility. A facility or part of a facility at which hazardous waste is intentionally placed into or on any land or water, and at which waste will remain after closure.

13. Energetic Wastes. Pertains to explosive and pyrotechnic wastes. A waste is considered an energetic waste if it is a reactive waste which:

(a) Is normally unstable and readily undergoes violent change without detonating;

(b) Is capable of detonation or explosive reaction when subjected to a strong initiating source or heated under confinement;

(c) Is readily capable of detonation, explosive decomposition or reaction at standard temperature and pressure; or

(d) Is a forbidden explosive as defined in 49 CFR 173.51, a Class A explosive as defined in 49 CFR 173.53, or a Class B explosive as defined in 49 CFR 173.88.

14. Explosive Contaminated Materials (ECM). Materials that have been in contact with explosives such as cardboard, floor sweepings, rags, wood, spent time fuzes, functioned items and metal, unless a significant amount of raw explosives are present as to pose an explosive hazard. Since ECMs do not meet the EPA definition of a reactive waste, they will not be considered a hazardous waste, and will be flashed at the Ammunition Burning Grounds for safety reasons only.

15. Explosive Hazardous Waste(EHW). A waste that exhibits the characteristics of reactivity primarily due to the presence of propellant, explosive or pyrotechnic components.

16. EPA Hazardous Waste Number. The number assigned by EPA to each hazardous waste listed in part 261, subpart D, of 40 CFR,

and to each characteristic identified in part 261, subpart C, of 40 CFR.

17. Flammable Liquid. A liquid with a flash point below 100°F (37.8°C).

18. Flammable Solid. A solid which will ignite readily or, under ordinary conditions, is liable to cause fires through friction or retained heat from manufacturing or processing, and which burn so vigorously that a serious transportation hazard could be created.

19. Generator. Any person, by site, whose act or process produces hazardous waste identified or listed in 40 CFR 261 or whose act first causes a hazardous waste to become subject to regulation.

20. Groundwater. Water below the land surface in a zone of saturation.

21. Hazardous Material. Any material intended for use in a process that is capable of producing adverse effects to human health and/or the environment

22. Hazardous Waste. A waste, which because of quantity, concentration, or physical, chemical, or infectious characteristics as defined in 40 CFR 261 may:

(a) Cause, or significantly contribute to, an increase in mortality or a serious irreversible, or incapacitating reversible illness; or

(b) Pose a substantial present or potential hazard to human health or the environment when improperly treated, stored, transported, disposed of or otherwise managed.

23. Hazardous Waste Generator. Any person or operation whose acts or process produces hazardous waste or whose act first causes a hazardous waste to become subject to regulation.

24. Ignitability. A solid waste exhibits the characteristics of ignitability (EPA Hazardous Waste Number D001) if a representative sample of the waste has any of the following properties:

(a) It is a liquid, other than an aqueous solution containing less than 24 percent alcohol by volume and has flash point less than 60°C (140°F), as determined by a Pensky-Martens

closed Cup Tester, using the test method specified in ASTM Standard D-93-79 or D-93-80 (incorporated by reference, see 40 CFR 260.11), or a Setaflash Closed Cup Tester, using the test method specified in ASTM Standard D-3278-78 (incorporated by reference, see 40 CFR 260.11), or as determined by an equivalent test method approved by the Administrator under procedures set forth in 40 CFR 260.20 and 260.21.

(b) It is not a liquid and is capable, under standard temperature and pressure, of causing fire through friction, absorption of moisture or spontaneous chemical changes and, when ignited, burns vigorously and persistently that is creates a hazard.

(c) It is an ignitable compressed gas as defined in 49 CFR 173.300 and as determined by the test methods described in that regulation or equivalent test methods approved by the Administrator under 40 CFR 260.20 and 260.21.

(d) It is an oxidizer as defined in 49 CFR 173.151.

25. Incompatible Waste. A hazardous waste which is unsuitable for:

(a) Placement in a particular device or facility because it may cause corrosion or decay of containment materials; or

(b) Commingling with another waste or material under uncontrolled conditions because the commingling might produce heat or pressure, fire or explosion, violent reaction, toxic dusts, mists, fumes, or gases, or flammable fumes or gases.

26. Indiana Administrative Code (IAC). The rules adopted and promulgated by the various state agencies that are published in a multi-volume set, containing all the rules made by all the agencies of Indiana State government.

27. Indiana Department of Environmental Management (IDEM). The governing body in the State of Indiana that has been granted authority, by the United States Environmental Protection Agency, to regulate various environmental matters, such as hazardous waste.

28. Laboratory Waste. Up to five gallons of any mixture of chemicals in known or approximate quantities that, after they have been used for their original purpose, are no longer needed. The classification of hazardous waste will apply when any of the characteristics described in the definition for hazardous waste

are applicable to the mixture and when it has been determined that there is no further use or marketability for the materials. This definition primarily applies to, but is not limited to, analytical testing operations.

29. Management or Hazardous Waste Management. The systematic control of the collection, source separation, storage, transportation, processing, treatment, recovery, and disposal of hazardous waste.

30. Material Safety Data Sheet (MSDS). A document required by 29 CFR 1910.1200, that accompanies a product, providing the handler with information on the compound's makeup, handling instructions, and potential hazards.

31. Operator. The person responsible for the overall operation of a facility/process.

32. Reactive Waste. A waste is considered reactive, as defined by 40 CFR 261.22, if:

(a) It is normally unstable and readily undergoes violent change without detonation;

(b) It reacts violently with water;

(c) It forms potentially explosive mixtures with water;

(d) When mixed with water, it generates toxic gases, vapors or fumes in a quantity sufficient to present a danger to human health or the environment;

(e) It is a cyanide or sulfide bearing waste, which when exposed to pH conditions between 2 and 12.5, can generate toxic gases, vapors or fumes in a quantity sufficient to present a danger to human health or the environment;

(f) It is capable of detonation or explosive reaction if it is subjected to a strong initiating source or if heated under confinement;

(g) It is readily capable of detonation or explosive decomposition or reaction at standard temperature and pressure; or

(h) It is a forbidden explosive as defined in 49 CFR 173.51, a Class A explosive as defined in 49 CFR 173.53, or a Class B explosive as defined in 49 CFR 173.88.

33. Resource Conservation and Recovery Act (RCRA). The Resource Conservation and Recovery Act (RCRA), which amended the Solid Waste Disposal Act, regulates the management of solid waste and hazardous waste. The Hazardous and Solid Waste Amendments (HSWA) of 1984 amended RCRA to include the cleanup, through corrective action, of releases of hazardous waste at RCRA-regulated facilities. RCRA requires cradle-to-grave management of hazardous waste through a record-keeping system that requires the manifesting of hazardous waste shipments from point of generation to ultimate disposal. Hazardous waste treatment, storage, and disposal facilities are regulated through the issuance of operating permits. RCRA provides that EPA may delegate authority to States to regulate hazardous waste under State law in lieu of RCRA. Irrespective of whether EPA has delegated hazardous waste authority to a State, State hazardous waste substantive and procedural requirements, including the requirement to obtain State permits, are applicable to Navy facilities under the Federal Facility Compliance Act (FFCA).

34. Release. Includes spilling, leaking, pumping, pouring, emitting, emptying, discharging, injecting, escaping, leaching, dumping, or disposing into the environment (including the abandonment or discarding of barrels, containers, and other closed receptacles) of any hazardous substance. IDEM reporting applies to releases that reach waterways, ditches and soil. IDEM does not consider a release to blacktop or concrete to be a spill if it does not run-off or penetrate, therefore reporting is not required.

35. Run-off. Any rainwater, leachate, or other liquid that drains over land from any part of a facility.

36. Run-on. Any rainwater, leachate, or other liquid that drains over land onto any part of a facility.

37. Sludge. Any solid, semi-solid, or liquid waste generated from a municipal, commercial, or industrial wastewater treatment plant, water supply treatment plant, or air pollution control facility exclusive of the treated effluent from a wastewater treatment plant.

38. Storage. The holding of hazardous waste for a temporary period, at the end of which the hazardous waste is treated, disposed of, or stored elsewhere.

39. Transport Vehicle. A motor vehicle or rail car used for the transportation of cargo by any mode.

40. Transportation. The movement of hazardous waste by air, rail highway, or water.

41. Transporter. A person engaged in the offsite transportation of hazardous waste by air, rail, highway, or water.

42. Treatment. Any method, technique, or process, including neutralization, designed to change the physical, chemical, or biological character or composition of any hazardous waste so as to neutralize such waste, or so as to recover energy or material resources from the waste, or so as to render such waste non-hazardous, or less hazardous; safer to transport, store, or dispose of; or make amenable for recovery, make amenable for storage, or reduced in volume.

ACRONYMS

ABG	Ammunition Burning Ground
AUL	Authorized Use List
CAAA	Crane Army Ammunition Activity
CERCLA	Comprehensive Environmental Response, Compensation and Liability Act
CFCs	Chlorofluorocarbons
CFR	Code of Federal Regulations
CSF	Central Storage Facility
CWA	Clean Water Act
DEMO	Demolition Range
DOD	Department of Defense
DOT	Department of Transportation
DRMO	Defense Reutilization and Marketing Office
EPCRA	Emergency Planning and Community Right-to Know Act
FFCA	Federal Facility Compliance Act
HM	Hazardous Material
HSWA	Hazardous and Solid Waste Amendments
HWPS	Hazardous Waste Profile Sheet
IAC	Indiana Administrative Code
IDEM	Indiana Department Environmental Management
LDR	Land Disposal Restrictions
MSDS	Material Safety Data Sheet
NSN	National Stock Number
NSWC-WSNO	NSWC Waste Stream Number
OB/OD	Open Burn/Open Detonation
PAT	Process Action Teams
PPO	Pollution Prevention Office
RCRA	Resource Conservation and Recovery Act
RQ	Reportable Quantity
SARA	Superfund Amendments and Reauthorization
TSD	Treatment, Storage and Disposal
USEPA	United States Environmental Protection Agency

HAZARDOUS WASTE MANAGEMENT PLAN

CRANE DIVISION, NAVAL SURFACE WARFARE CENTER

CRANE, INDIANA 47522-5009

AMENDED AUGUST, 1999

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CHAPTER 1
REQUIREMENTS FOR GENERATORS

1.1 APPLICATION FOR HAZARDOUS WASTE GENERATION APPROVAL

1.1.1. Any operation of NAVSURFWARCENDIV Crane using hazardous materials must be evaluated to determine if the process will result in the generation of solid or hazardous waste (Specific Federal Regulations can be found in Appendix 1). If it has been determined that a waste will be generated, careful consideration of the processes involved should produce an approximation of the amount of waste to be generated.

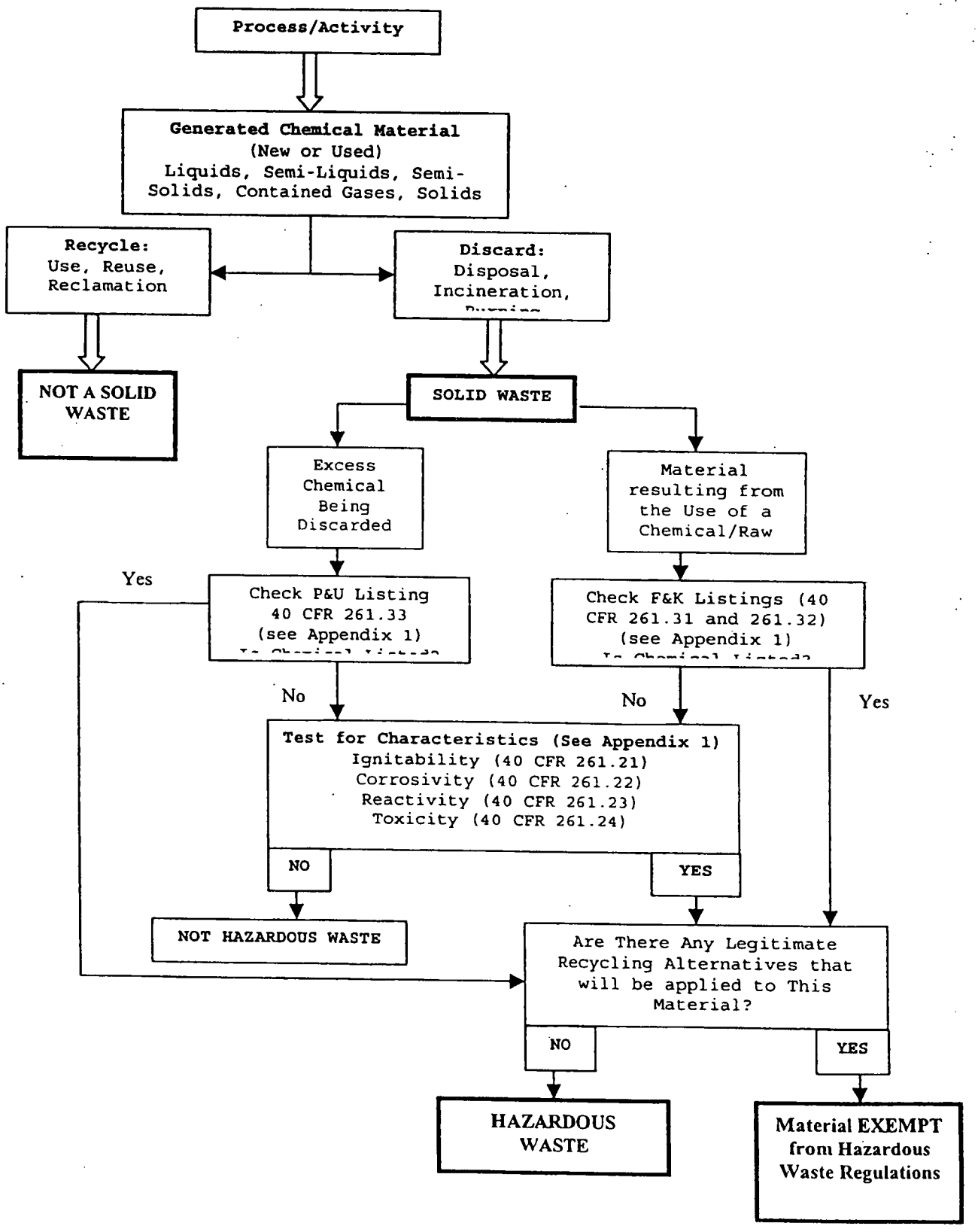
1.1.2. The following flow diagram (Figure 1) on page 1-2 should be used as a guide to determine whether the waste will be classified as "hazardous." If, after careful review of this diagram, it is determined that the process residue or waste may be hazardous, then the generating code or activity must complete Section 1 of the Hazardous Waste Profile Sheet (HWPS), found in Appendix 2, and return it to the Environmental Protection Department (EPD). Blank copies of the HWPS should be obtained from the EPD, B-3260 by calling 854-1132. The EPD staff will assist in the preparation of Section 1, upon request.

1.1.3. With the information provided in Section 1 of the HWPS, the EPD staff will evaluate and characterize the material(s) involved and complete Sections 2 and 3.

1.1.4. A completed copy of the HWPS will be returned to the originator. A NAVSURFWARCENDIV Crane Waste Stream Number (NSWC-WSNO) will be recorded at the top of the HWPS, and again in Section 3. This number will be unique to the waste stream listed in Section 1, as well as to the code or activity providing the information. Absolutely no changes or modifications are to be made in the composition of the waste material without notification to the EPD. If processes or material changes occur, then a new HWPS must be completed. The EPD should be contacted immediately regarding changes that have occurred, along with the NSWC-WSNO that was affected.

1.1.5. If the waste has been determined to be hazardous, the information found in Section 3 of the HWPS will be used to properly complete the container labeling requirements found in section 1.6.4.

Figure 1. Guide to determine if waste is hazardous.



NOTE

> **Placing hazardous waste labels on containers without the profiling process is strictly prohibited. Absolutely no hazardous waste labels will be placed on containers unless this process has been completed or an EPD Representative has made the determination and provided special instructions.**

With this in mind, as soon as it has been determined that a hazardous material will be used in a process, and prior to generation, the profiling process should be initiated. In addition, if a process does not use a hazardous material, but involves mechanical removal of components that become wastes, then the generator would also need to initiate profiling.

1.1.6. The EPD will provide the generator with a uniform logbook for record-keeping and site management. The operator of each hazardous waste generating process will provide the following equipment and items, in order to comply with 40 CFR 265.32 -. 34. The EPD will be available, upon request, to offer assistance with these requirements:

1.1.6.1. Required Equipment: All facilities must be equipped with the following, unless hazards posed by the waste handled at the facility do not specifically require a particular kind of equipment listed below:

a. An internal communications or alarm system, capable of providing immediate emergency instruction (voice or signal) to facility personnel;

b. A device, such as a telephone or a hand-held two-way radio, capable of summoning emergency assistance from NSWC police and fire departments. This device should be immediately available at the scene of operations;

c. Portable fire extinguisher(s), fire control equipment (including special extinguishing equipment, such as that using foam, inert gas, or dry chemicals), spill control equipment, and decontamination equipment; and

d. Water at an adequate volume and pressure to supply water hose streams, foam producing equipment, automatic sprinklers, or water spray systems.

1.1.6.2. Testing and Maintenance of Equipment: Facility communications or alarm systems, fire protection, spill control, and decontamination equipment, where required, must be tested and maintained, as necessary, to assure proper operation in time of emergency.

1.1.6.3. Access to Communications or Alarm Systems: Whenever hazardous waste is being poured, mixed, spread, or otherwise handled, all personnel involved in the operation must have immediate access to an internal alarm or emergency communication device, either directly or through visual or voice contact with another employee, unless such a device is not required under Section 1.1.6.1. If there is ever one single employee on the premises while the facility is operating, he must have immediate access to a device, such as a telephone or a hand-held two-way radio, capable of summoning external emergency assistance, unless such a device is not required under Section 1.1.6.1.

1.1.6.4. A Site Plan Drawing (8.5" x 11"): This drawing should show where waste is accumulated, the location of the spill equipment, the fire alarm and the telephone.

1.1.6.5. A Spill Plan: This plan should meet the requirements of reference (g). The Spill Plan shall be specific to the generation site and based on the types of hazardous material used in the generating process. It must include who to notify and what actions will be taken.

1.2. WASTE ACCUMULATION SITE MANAGEMENT PLANS

1.2.1. Waste accumulation sites must be maintained and operated to minimize the possibility of a fire, explosion, or any unplanned sudden or non-sudden release of hazardous waste or hazardous waste constituents to air, soil, or surface water, which could threaten human health or the environment. Furthermore, supervisors of operations that generate hazardous waste should familiarize themselves and comply with the requirements of reference (g).

1.2.2. **Type I Accumulation Point:** A Type I Accumulation Point is satellite accumulation of up to 55 gallons of hazardous waste, or up to 1 quart of acutely hazardous waste (see Appendix 1 for list of acutely hazardous wastes), at or near the point of generation, as defined in 40 CFR 262.34(c). This type of management will be used for operations that are determined to generate small volumes of a single waste stream regularly and at

a rate that will not fill a 55 gallon container within ninety days. A container selection smaller than a 55 gallon drum will be made. This will prevent exceeding the 55 gallon limit. For example, if a 30 gallon drum is used to accumulate the waste and it becomes full, a call can then be placed to have the waste picked up and the operation can continue by starting a second container. A quantity violation will not exist if the container is not removed within three days since the volume at this location has not exceeded the 55 gallon limit. If the full container is not moved by the time the second 30 gallon container becomes full, then there is a quantity violation of approximately five gallons. If the first full container is called in for pickup as soon as the second container is started, the first container will be removed before a quantity violation occurs.

NOTE

If the generating process also has an approved Type II, 90 day storage area (see section 1.2.3. for definition), the full container can be moved to that area. The date the container became full will be placed on the label. The appropriate entry will be made in the logbook and called in for pickup. The fill date becomes the date that will start the 90 day limit no matter what the size of the container. There are only a few operations that will be set up for this type of management.

1.2.2.1. Ensure that the waste is placed in a non-leaking container of good condition. If the container is not in good condition, or begins to leak, the hazardous waste is to be transferred to a container that is in good condition;

1.2.2.2. Use a container made of, or lined with, materials that will not react with, and are otherwise compatible with, the hazardous waste to be stored, so that the ability of the container to contain the waste is not impaired;

1.2.2.3. Always completely close the container holding hazardous waste during storage, except when it is necessary to add or remove waste and in accordance with section 1.2.2.4;

1.2.2.4. Not open, handle, or store a container holding hazardous waste in a manner that may rupture the container or cause it to leak; and

1.2.2.5. Label the containers in accordance with the requirements of paragraph 1.6.4.

NOTE

It is mandatory that the fill date be placed on the label at the onset of the excess accumulation. It is imperative that turn-in be made promptly. Waste in excess of 55 gallons must be removed from the site to the Central Storage Facility (CSF) or approved Type II, 90 day storage site within three days of final fill date.

1.2.2.6. Procedures for waste turn-in are found in subparagraph 1.6.7.

NOTE

The requirement to perform and document weekly inspections of a Type I site has been removed from this instruction. The site will be subject to random compliance inspections.

1.2.3. **Type II Accumulation Area:** A Type II Accumulation Area is accumulation of quantities of hazardous waste greater than 55 gallons for up to 90 days, as defined in 40 CFR 262.34(a). The operator of a Type II Accumulation Area must:

1.2.3.1. Ensure that the waste is placed in a non-leaking container of good condition. If the container is not in good condition, or begins to leak, the hazardous waste is to be transferred to a container that is in good condition;

1.2.3.2. Use a container made of, or lined with, materials that will not react with, and are otherwise compatible with, the hazardous waste to be stored, so that the ability of the container to contain the waste is not impaired;

1.2.3.3. Always completely close the container holding hazardous waste during storage, except when it is necessary to add or remove waste;

1.2.3.4. Not open, handle, or store a container holding hazardous waste in a manner that may rupture the container or cause it to leak;

1.2.3.5. Locate the containers holding ignitable or reactive waste at least 50 feet (15 meters) from the building and/or the facility's property line;

1.2.3.6. Not place incompatible wastes, or incompatible waste and materials in the same containers;

1.2.3.7. Not place hazardous wastes in an unwashed container that previously held an incompatible waste or material;

1.2.3.8. Separate storage containers holding hazardous waste that are incompatible with other wastes or materials stored nearby, from the other waste or materials by means of a dike, berm, wall or other device, and liquid wastes must have secondary containment; and

1.2.3.9. Label the containers in accordance with the requirements of paragraph 1.6.4.

NOTE

It is mandatory that the date each period of accumulation begins is clearly marked and visible for inspection on each container.

1.2.3.10. Waste which presents the possibility of fire, explosion, or any unplanned sudden or non-sudden release must be separated and protected from sources of ignition or reaction including, but not limited to: Open flames, smoking, cutting and welding, hot surfaces, frictional heat, sparks, radiant heat, etc. "No Smoking" signs must be conspicuously placed wherever there is a hazard from ignitable or reactive waste;

1.2.3.11. Maintain an adequate aisle space (i.e., 30 to 39 inches) around the hazardous waste containers in order to allow the unobstructed movement of personnel, fire protection equipment, spill control equipment, and decontamination equipment to any area of the Type II Accumulation Area in an emergency; and

1.2.3.12. Phone the EPD to have wastes moved to Building 2993, the permitted CSF. Procedures for waste turn-in are found in subparagraph 1.6.7; and

NOTE

It is imperative that turn-in is made promptly. Waste is to be moved from the Type II storage area to the CSF or to the disposal facility within ninety days from the Fill Date placed on the label. If using the Type II site for the purpose of satellite accumulation, the "start date" will be used to determine the ninety day period.

1.2.3.13. Type II Accumulation Area will be inspected weekly, using the Waste Accumulation Weekly Inspection Checklist found in Appendix 3. Copies of the completed forms will be kept in the site operation logbook.

NOTE

The requirement to forward a copy of the Waste Accumulation Weekly Inspection Checklist to the EPD has been removed from this instruction. The documented inspection is required as a condition to operate. Failure to perform this inspection will be a violation.

1.3. MANAGEMENT OF LABORATORY HAZARDOUS WASTES AND POTENTIALLY USABLE UNWANTED MATERIALS

1.3.1. Laboratory wastes include five gallons or less of any mixture of chemicals in known or approximate quantities that, after they have been used for their original purpose, are no longer needed. The classification of hazardous waste will apply when any of the characterizations described in the definition for hazardous waste are applicable to the mixture. This definition primarily applies to analytical testing operations, but may also be applicable for partial containers of Hazardous Material that have no further use and are not marketable.

NOTE

Unwanted excess hazardous materials do not necessarily make a hazardous waste.

1.3.2. Laboratory wastes, that have been determined to be hazardous wastes, must be grouped for compatibility and marked

with the words "Hazardous Waste", the date of the waste determination and a description of the hazardous material. The determination will normally be based on the material having a potential for further use. Options for further use may include transfer to another process that is on the Authorized Use List (AUL) for the same material. If acceptable, return to the Haz-Mat center to be restocked or have the material screened for potential sale through DRMO. In most cases the manufacturer's label can be referred to for best description. Only after these considerations have been made should a hazardous waste label be applied to an unopened container of hazardous material that has potential for reuse. The size of the label may vary due to the size of the container. Efforts must be made to not apply labels over important manufacturer's label information. The EPD can provide small container labeling assistance. Most departments have label making software that is capable of producing labels that will meet the size requirement. Small vials, bottles, or cans can be grouped according to hazard class and compatibility, one label can be used to represent the group. The group can be housed in a larger container or a lab waste collection pan, which is available from the EPD. Small laboratory waste vials may also be placed in a zip lock bag, with the hazardous lab waste label affixed to the outside of the bag. The EPD will supply directions and assistance to complete the waste label if requested.

A Laboratory Waste Inventory Form, found in Appendix 4, must be completed and submitted to the EPD for collection of such wastes.

NOTE

Material in original containers, which can be supported with a Material Safety Data Sheet (MSDS), will not be accepted by the EPD, until such material has gone through the previously described decision process.

1.4. MANAGEMENT OF ENERGETIC WASTES AND RESIDUE

1.4.1. Explosives, pyrotechnic, and propellant operations that produce, manufacture, store, or test items of an energetic nature, have the potential to generate wastes that are considered reactive or ignitable hazardous waste. These operations must meet all requirements of NAVSEA OP 5 Volume 1,

Sixth Revision (reference (f)). Specifically, all of Chapter 2 and paragraph 4-1.2.2(c) should be reviewed.

1.4.2. In order to comply with 40 CFR 265.17, generators of energetic wastes, or wastes contaminated with energetic materials (such as solvents) must:

1.4.2.1. Take precautions to prevent accidental ignition or reaction of ignitable or reactive waste. Such wastes must be separated and protected from sources of ignition or reaction, including but not limited to: open flames, smoking, cutting and welding, hot surfaces, frictional heat, sparks, spontaneous ignition and radiant heat. While ignitable or reactive wastes are being handled, smoking and open flames must be confined to specially designated locations. "No Smoking" signs must be conspicuously placed wherever there is a hazard from ignitable or reactive waste; and

1.4.2.2. The treatment, storage or disposal of ignitable or reactive wastes, and the mixture or co-mingling of incompatible wastes, or incompatible wastes and materials, must be conducted so that it does not:

a. Generate extreme heat or pressure, fire, or explosion, or violent reaction;

b. Produce uncontrolled toxic mists, fumes, dusts, or gases in sufficient quantities to threaten human health;

c. Produce uncontrolled flammable fumes or gases in sufficient quantities to pose a risk of fire or explosions;

d. Damage the structural integrity of the device or facility containing the waste; or

e. Through other like means threaten human health or the environment.

1.4.3. Operations that generate explosive waste or pyrotechnic scrap must have a designated storage location designed to comply with NAVSURFWARCENDIV Crane fire, safety, and environmental requirements and as specified in Chapter 11 of reference (f).

NOTE

This site must be managed as a hazardous waste Type I-X Accumulation Area, as provided in Appendix 5 of this plan, unless otherwise instructed by the EPD or as specified in Standard Operating Procedures. A Type I-X Accumulation Area is defined as a Type I Accumulation Point, except that it applies to explosive hazardous waste. A Type II-X Accumulation Area is defined similarly as a Type II Accumulation Area, but also applies only to explosive hazardous waste.

1.4.4. In addition to the aforementioned requirements in this section, the following are additional NAVSURFWARCENDIV Crane requirements. The generator must:

1.4.4.1. Make every effort, at the process line or generating activity, to keep the quantity of waste to an absolute minimum, and to separate the waste from incompatible materials;

1.4.4.2. Properly close the container used to collect the waste and affix the Explosive Hazardous Waste Scrap label (see Appendix 6). **Do not tighten the container seal such that its contents could cause a rupture or release.** Containers must be properly labeled and should include the volume of material in each container; and

1.4.4.3. At a minimum of once per day, move all explosive waste or pyrotechnic scrap from the process, or generating location, to the designated storage location, to await movement to the treatment site. An exception to this requirement could be granted, but it must be documented in the applicable SOP. In no case shall quantities greater than 55 gallons be held for more than three days without being taken to the treatment site.

1.4.4.4. To be in compliance, the following standards must be met.

a. Waste must be placed into a container in good condition.

b. The container must be made of materials compatible to the waste being collected.

c. The container must be placed at or near the point of generation and in an area approved by the EPD. The storage areas holding Explosive Hazardous Waste must also be approved by Explosive Safety.

d. Secondary containment must be provided for any containers that are holding liquids.

e. The storage/collection area must also provide protection from precipitation run-on, run-off and accumulation.

f. The storage/collection area must be under the operator's control.

g. Signs must be posted at the storage/collection area. Required signs include the following: "No Smoking" and the words "Explosive Hazardous Waste" must be displayed at the entrance to the storage/collection area. An example of a sign to be posted at the storage/collection area is provided in Appendix 7.

h. The operator must maintain records that track the materials into the storage location and to the next management facility. In most cases this will be the Ammunition Burning Ground (ABG) or the Demolition Range (DEMO).

i. Additional record keeping required includes a site diagram that displays the storage location, location of emergency response equipment and the location of any communication devices that may be used to summon assistance in the case of emergency. A list of phone numbers and/or radio call signs must be provided and maintained with the communication device.

1.4.5. A Contaminated Scrap Manifest form (found in Appendix 8) must be prepared for each explosive hazardous waste as it is moved from the production, processing or testing area to the designated storage area awaiting transfer to the treatment facility, ABG or DEMO. The Contaminated Scrap Manifest will be used as the required Explosive Hazardous Waste generation log as well as the tracking document to assure the material is properly disposed of. The manifest should be completed as follows:

1.4.5.1. The Contaminated Scrap Manifest form has five colored pages. If a generating process has wastes

designated for two separate treatment facilities, a separate manifest must be prepared for each shipment, per facility. Also, the generator should complete the treatment facility block on the form. The completed Contaminated Scrap Manifest or Manifests that clearly describes and accounts for the type and volume of materials in storage awaiting disposition will be kept in the generators log book.

1.4.5.2. When the transporter arrives, the supervisor, or designated representative, must provide the completed scrap manifest to the transporter for his signature in the appropriate block. After receiving the transporter's signature, the generator should remove the pink copy and retain it for his records and send the gold copy, within five working days, to the EPD.

1.4.5.3. The transporter will then deliver the material to the designated facility. Upon arrival at the facility, the facility operator will compare the manifest with the material. If no discrepancies are found, the facility operator will sign and date the remainder of the forms in the appropriate block. At this point, the transporter will remove the yellow copy and return it to the EPD. The facility operator will retain the green copy for the treatment facility's files, and return the white copy to the EPD.

1.4.6. The supervisor must arrange to move the material to the designated treatment site within the time period established in applicable Standard Operating Procedures (SOPs). Transportation is provided by the operators of the treatment facilities, but must be coordinated. Some operations may deliver materials, provided that all necessary arrangements have been made with the treatment facility managers, and the designated transporters are qualified and certified to do so.

1.4.7. Prior to the generation of waste, the operation supervisors and managers are responsible for ensuring that the waste can be properly managed at one of the NAVSURFWARCENDIV Crane treatment facilities. Crane treatment facilities include the ABG and DEMO. Contact the EPD if help is needed to determine the proper treatment facility to utilize.

1.4.8. Disposition of Test Residue and Spent Munitions Hardware. All residues from the testing of ordnance items that have properly functioned but still may require flashing in accordance with the conditions found in OP 5 paragraph 13-18.1 shall be labeled and managed as described in this section. The

types of items may include residues from propellant, explosive or pyrotechnic (PEP) testing as well as casings and hardware from spent munitions operation items. The designated area to stage these items must be marked with a sign (see Appendix 9 for a copy of the sign and Appendix 10 for a container label) displaying the following words:

SPENT MUNITIONS ITEMS

.Per NAVSEA OP 5 Para.13-18.1
.Items that require flash burning shall be marked, "OP 5 items awaiting safety flashing".
.Note: Items that do not require additional flashing should be marked "INERT" and transferred for scrap metal recovery or disposed of as solid waste.

1.4.9. The labels and assistance with signs can be obtained through the EPD. These items should be frequently transferred to the Ammunition Burning Grounds for flash burn treatment. A Contaminated Scrap Manifest shall be used to document inventory and transfer. While these items are being stored at the testing site, they should be protected from precipitation run-on, run-off.

1.5. MANAGEMENT OF BATTERIES

1.5.1 Crane Division, Naval Surface Warfare Center and its tenant activities are engaged in many diverse programs and projects that result in excess and/or unwanted batteries. For the purpose of definition, the following two categories should describe most operations or functions that result in excess batteries.

NOTE

The USEPA has issued the "Universal Waste Rule", which is designed to reduce the amount of hazardous waste items in the municipal solid waste stream, encourage recycling and proper disposal of certain common hazardous waste and reduce the regulation on businesses that generate these wastes. Batteries are identified as one of the wastes covered by this rule. This rule will allow for some modification to the handling practices that may vary from other hazardous waste management requirements.

1.5.1.1. Category I: End User or Consumer. This would, for example, include batteries that are expended through

normal use in tools, flashlights and office equipment. This is a type of battery that has served its original purpose and is replaced by the user when needed.

1.5.1.2. Typically, Category I End users or consumers will generate batteries that are of cell sizes commonly referred to as AAA, AA, C and D. The solid waste recycling contractor has placed small blue collection boxes at locations throughout the Crane Division base. Category I generators can place batteries of the previously described sizes in these containers to be collected for recycling (on a basis as frequent as the recycled paper collection). This option is limited to Alkaline and Nickel Cadmium (Ni-Cad). For batteries of other chemical composition, such as mercury and lithium, generators must contact the EPD for collection.

1.5.1.3. Category II: Industrial Process Generator. This category is used to describe processes that involve demilitarization, refurbishment or removal of batteries from equipment being declared excess. Each type of these processes usually involves the removal and management of batteries that are consistent in size and chemical composition.

1.5.1.4. Operations that are classified as Category II must refer to and comply with Sections 1.1 through 1.1.5. of this Instruction. Those sections refer to the process used to prepare and submit a Hazardous Waste Profile Sheet (HWPS). Detailed information regarding the size and chemical composition of the batteries must be provided and attached to the HWPS when submitted to the EPD. After the HWPS has been reviewed, Sections 2 and 3 of the HWPS will be completed by the EPD and returned to the generator. Special instructions will be included that will provide marking/labeling, storage and turn-in guidance. When batteries are being turned in to the DRMO for management, the Defense Logistics Agency Hazardous Waste Profile Sheet (Appendix 11) must be completed.

1.6. GENERAL CONTAINER REQUIREMENTS

All containers must be approved by the EPD for the collection of each specific waste material.

1.6.1. Selection of Containers: The EPD will evaluate the process generating the waste and review the estimated rate of generation. With this information, they will determine what type of container is compatible with the specific waste. The smallest possible container will be selected to support the

project in the regulated site. This means of container management will ensure that the most compliant and cost effective methods will be maintained.

1.6.2. Availability of Containers: Containers are available from the EPD. The type of containers to be used in an operation will be stated in the hazardous waste profile process (see Section 1.1).

1.6.3. Reuse of Containers: With approval from the EPD, it may be determined that the collection of spent materials into their original shipping containers may prove to be both compliant and cost effective.

NOTE

This must be approved by the EPD.

1.6.4. Container Labeling: All contained wastes must be properly labeled by the generator. This should include the exact contents of the waste, along with specific concentration, if known. Labels and permanent markers are available from the EPD. The name used for a process waste shall be used consistently for that waste throughout its generation, as long as the process, or its components, does not change. The established name shall be used for obtaining Hazardous Waste Generation Approval. The name shall not be changed without written request to and approval from, the EPD. Compliance with this policy will eliminate costly re-sampling and analysis, and will possibly reduce the risk of rejection at their ultimate disposal sites.

NOTE

Placing hazardous waste labels on containers without first completing the profiling process or having clearly made a determination that the residues or material is in fact a hazardous waste is strictly prohibited. Absolutely no hazardous waste labels will be placed on containers unless this process has been completed. This can often cause a non-compliant condition that can result in a regulatory violation. If it is uncertain as to what the characterization of materials should be, the container should be clearly marked as to what the material is and immediately seek assistance from the EPD office.

1.6.4.1. Hazardous Waste Labels: The Hazardous Waste label can be found in Appendix 12. Remove the backing from the label and attach the label to the container of waste. All portions of the label must be filled out in permanent ink. Writing must be plain and readable. The information placed on the label will be used for departmental logging and record keeping in the Hazardous Waste Generation Log (see Appendix 13). The following information must be filled in by the generator:

a. Waste Description: This information can be found in section 3 of the HWPS;

b. NSWC Waste Profile Number: This information can be found in Section 3 of the HWPS;

c. Contact: This is to include the name of the person most knowledgeable of the waste's history and chemical composition. The Project or Building Supervisor is preferred;

d. Extension: Provide the extension number of the contact;

e. Start Date: This is the date the first increment of waste is placed in the container;

NOTE

It is critical that the start date is accurately filled in on the label for a Type II site. This data starts the ninety day time period.

f. Fill Date: When the level of material reaches approximately three to four inches from the top, the container is to be closed and that day's date is to be placed on the label for a Type I Accumulation Point site. This date means volumes greater than 55 gallons must be moved to the CSF or Type II site within three days.

1.6.4.2. Non-Regulated Waste Labels: If, through the approval process, the material is determined to be non-hazardous or non-regulated, then the Non-Regulated Waste label, found in Appendix 14, will be applied to the container. Remove the backing from the label and attach the label to the container of waste. All portions of the label must be filled out in permanent ink. Writing must be plain and readable. The information placed on the label will be used for departmental logging and record keeping in the Hazardous Waste Generation

Log. The following information must be filled in by the generator:

a. Waste Description: This information can be found in Section 3 of the HWPS;

b. NSWC Waste Profile Number: This information can be found in Section 3 of the HWPS;

c. Contact: This is to include the name of the person most knowledgeable of the waste's history and chemical composition. The Project or Building Supervisor is preferred;

d. Extension: Provide the extension number of the contact;

e. Start Date: This is the date the first increment of waste is placed in the container; and

NOTE

It is critical that the start date is accurately filled on the label.

f. Fill Date: When the level of material reaches approximately three to four inches from the top, the container is to be closed and that day's date is to be placed on the label.

1.6.4.3. If the information provided in the HWPS is too vague to clearly determine that the material is a hazardous waste, then the Non-Regulated Waste label will be applied to the container. The material will undergo laboratory analysis and if, through the analysis, the material is determined to be hazardous, the Non-Regulated Waste label will be replaced with a Hazardous Waste label. This will not be accomplished until the analytical data has been received.

1.6.5. **Handling of Empty Containers:** Each generator will make every reasonable effort to fully use the contents of each container. They are to ensure that the total residue remaining in the container equals less than one inch.

NOTE

Containers, which hold a minimum of one inch of residue, and containers, which have been used to maintain any of the acute hazardous wastes found in Appendix 1, are considered to be a hazardous waste. Prior to disposal, the generator must purge the residue from the container, then clean the container by triple rinsing. If this is not accomplished, the container is to be disposed of as hazardous waste. Triple rinsing requires the use of a solvent capable of removing the residue from the container. The EPD should be consulted on what to use for triple rinsing. A specific quantity of solvent, equal to 10 percent of the container capacity, must be used for each of the three rinses. After rinsing, the solvent must be containerized as a hazardous waste, and disposed of according to hazardous waste requirements. Future utilization of these materials should be closely evaluated and substitutions for less hazardous materials should be made, if possible.

1.6.5.1. A container of material, other than the acute hazardous wastes listed in Appendix 1, with less than one inch of residue may be disposed of as an empty container, if disposal is necessary.

NOTE

All free flowing liquids must be removed when possible.

1.6.5.2. A container that has held a compressed gas is considered empty when pressure in the container approaches atmospheric pressure and no longer expels any product. At this time, the container can be disposed of as an empty container, if disposal is necessary. Compressed gas cylinders that are refillable must be returned to the vendor. If the cylinder is not leased and no longer needed, it must be empty and have the valve neck open (fittings removed) before the DRMO will accept the item as scrap metal. Contact the EPD for assistance with compressed gas cylinders when the contents and/or pressure are uncertain.

1.6.5.3. Empty containers are to be turned in to DRMO using DD Form 1348-1. Empty containers are not to be accumulated. If it is necessary to store empty containers near the work site, they must meet all requirements of this section. Furthermore, empty containers must be sealed with their caps in place, marked as empty, and stored on their sides. This process will prevent the accumulation of precipitation on top of the container and will help to maintain the integrity of the container.

1.6.6. Placement and Inspection of Containers in Use: The generator must inspect areas where containers are stored weekly. They are to verify the absence of leaking containers, and they are to examine containers for deterioration caused by corrosion or other factors. All containers used for waste storage must immediately be documented on the Hazardous Waste Generation Log (see Appendix 13).

1.6.7. Procedures for Waste Turn-In: When a container at the waste accumulation site is ready to be transferred to the Center's permitted CSF, the generator will call the EPD (extension 1132 or 3114). The generator will be asked to furnish the hazardous waste profile number, the waste label number, and the quantity to be picked up. At this time, if a charge number has not already been established with the EPD for the management of the waste, then one will be issued. The EPD will provide the generator with a four-digit call number, which the generator is to record in the operation logbook. Along with the call number, the generator is to record the date of the call.

CHAPTER 2
REQUIREMENTS FOR THE ENVIRONMENTAL PROTECTION DEPARTMENT

2.1. TRANSPORTATION

2.1.1. Transportation from Accumulation Sites: Transportation from the accumulation site is completed by the EPD Hazardous Waste Handler in accordance with the applicable SOP. Upon arrival at the site, information concerning the pick-up is provided to the Hazardous Waste Handler by the generator. The Hazardous Waste Handler will check the information on the Hazardous Waste Program Control Record (see Appendix 15) against the information on the container's label. If any discrepancy is found that would cause the container to be improperly transported or stored, the Hazardous Waste Handler will inform the generator immediately. Any problems will be corrected upon notification. Wastes will not be transported until the problem has been corrected.

If drums are transported on pallets, a ratchet nylon strap of appropriate size will be placed around the girth of the drums to prevent movement on the pallet. Another strap will be placed around the top of the drums and secured to the bed of the truck to prevent shifting of the pallet. Normally, drums are to be placed on the floor of the truck and moved all the way to the front of the bed. A nylon ratchet strap will be fastened to the front stakes of the bed, around the girth of the rear drums, and tightened to prevent movement of the drums during the transport. Containers will be unloaded from the delivery vehicle using a hydraulic tailgate. Containers will be moved into storage by a forklift with a drum cradle, or by a hand dolly. The wheels of the delivery vehicle will be chocked during the unloading process. Drums will be placed on pallets with the forklift and drum cradle and then positioned by hand. Palletizing the drums will be accomplished on the smooth surface of the asphalt pad outside the CSF. Pallets will then be moved into storage by a forklift or palletjack.

2.1.2. Transportation for Disposal: Hazardous Waste stored at the CSF will be transported, by a contractor, to an off-site disposal facility. Prior to pickup, the EPD will ensure that the waste is in an approved container, the container is properly sealed, and the container is properly labeled as to its contents.

2.2. BUILDING 2993 PERMITTED CENTRAL STORAGE FACILTIY (CSF)

Building 2993 is the single destination for hazardous wastes to be stored at NAVSURFWARCENDIV Crane (for periods that may exceed 90 days), prior to being transported to an approved off-site RCRA permitted facility. Additional requirements for the management of wastes at the CSF are found in the facility's Final RCRA Part B Permit.

2.2.1. Container Storage Procedures: Containers will be stored in a manner that does not allow any possibility of puncture by contact with other containers. Wastes in glass bottles, jars, or jugs will be placed in plastic or metal trays to minimize the possibility of breakage or spills. Glass containers will not be stacked unless placed in DOT approved over-pack cartons. A minimum aisle space of 2.5 - 3 feet will be maintained around drums, to allow the unobstructed movement of personnel, fire protection equipment, forklift, spill control equipment, and decontamination equipment. Each drum will, at all times, be positioned so that the hazardous waste label is clearly visible.

Containers holding free liquids will be stored on pallets that can be moved with a forklift, or similar equipment. Up to four 55-gallon drums may be placed on one pallet. Pallets that do not permit contact between adjacent drum rims will be used, if drums are stacked. Containers may only be stacked two high. Pallets will be inspected for nails (if wooden) and the condition of welds (if metal). Any pallet found to be unsound in structure will be discarded, unless safe repairs can be made.

2.2.2. Container Identification: Each hazardous waste container in the CSF will be properly labeled to include contents, dates in storage, and NSWC-WSNO. Faded writing will be rewritten with a permanent marker. Labels in poor condition will be replaced with a new label. Information on the old label will be transferred to the new one.

2.2.3. Storage Records: When a container is placed in storage at the CSF, the Central Storage Facility Log Sheet (see Appendix 16) must be completed by the waste handler and turned in daily. Information placed on the CSF Log Sheet includes the four-digit number found on the label, the NSWC-WSNO, the weight of the container, the date received at the CSF, and the location in which the container is stored. Upon receipt of the CSF Log Sheet, the EPD staff will transfer the information to their computer database.

2.2.4. Spill/Leaks: Hazardous waste spills and leaks will be cleaned up promptly at the CSF. Spill material will be disposed of as hazardous waste. Residues from spill will be packaged and labeled, as appropriate for the spill material. Waste handlers executing spill procedures will immediately report the spill to the EPD Manager.

2.2.5. Repack Leaking/Damaged Containers: Leaking or damaged hazardous waste containers will be repackaged into Department of Transportation (DOT) recover drums. If this procedure is not feasible, the contents will be placed into an approved container and the damaged container will be handled as hazardous waste. The new container will be correctly packaged and labeled with the information from the original leaking container.

2.2.6. Inspection: Containers will be inspected at the CSF on a weekly basis concerning their condition. These inspections will ensure that caps and lids remain closed, except when transferring wastes. Integrity of safety equipment and spill control materials will also be included in the weekly inspections. Inspections will be recorded in the Central Storage Facility Weekly Inspection Log, (see Appendix 17).

2.3. PERMITTED TREATMENT FACILITIES

Crane treatment facilities include the Ammunition Burning Grounds, Old Rifle Range and the Demolition Range. These operations must comply with the container management and inspection requirements outlined in the Part B Permit and the interim status Subpart X Permit.

2.4. TRAINING

As a generator of hazardous waste, and as an operator of a Treatment, Storage, and Disposal (TSD) facility, Crane Division is required to have a written training plan for personnel involved with hazardous waste management. These personnel are required to be familiar with the regulations for waste management and spill response activities, and to be able to handle hazardous waste in a safe manner.

2.4.1. General Requirements

2.4.1.1. All Directorate Heads and Heads of Tenant Activities will:

a. Ensure that all personnel, involved in any aspect of hazardous waste management (handlers, operators, production supervision, procedure writers, process engineers, demilitarization facility personnel, transporters, etc.), attend an introductory hazardous waste management course within six months of their initial assignments to duty, and attend annual refresher sessions thereafter;

b. Whenever possible include all related hazardous waste assignments in the position descriptions of pertinent personnel; and always

c. Maintain, on file, the names of employees who work with hazardous waste, and include work location, job title, position start date, successful training documentation (including on-the-job training), and any documentation regarding reasons why an employee has not attended such training (e.g., illness, detail to different position, etc.).

2.4.1.2. The Environmental Protection Department will:

a. Ensure that training is coordinated with Supply Directorate and Employee Services Directorate; and

b. Make training available as necessary

2.4.1.3. Employee Services will:

a. Ensure that all classroom instructions (on and off Center) are recorded in each individual's training record within the computerized Training Information Management System report and/or the Explosive Certification Record, as applicable. This form of monitoring will assure that all required personnel have been notified and have received all necessary training;

b. Maintain training records of all current personnel, until closure of the facility; and

c. Maintain training records of former employees, for at least three years from the date the employee last worked at the facility.

2.4.2. The list, which follows, includes the various types of training required by this plan, along with a brief description of the training elements involved. It is the responsibility of all Directorate Heads and Heads of Tenant

Activities to determine the type(s) of training necessary for each employee.

2.4.2.1. Hazardous Waste Generator Training: Employees who manage waste, associated with a Type I and/or Type I-X Accumulation Point or a Type II and/or Type II-X Accumulation Area must attend the Hazardous Waste Generator training. Training will include containerization management practices at the Type I, Type I-X, Type II or Type II-X site. Requirements of this instruction, other applicable written hazardous waste procedures, and written standard operating procedures will also be included in the training. Training should include a 2-hour initial session, followed by annual refresher courses.

2.4.2.2. TSD Worker: [29 CFR 1910.120 (p) (7) and 40 CFR requirement] Employees working at ABG, DEMO, Old Rifle Range, and/or Building 2993 must attend a 24-hour initial course, plus an annual 8-hour refresher.

2.4.2.3. Spill Response Operations Level: [29 CFR 1910.120 (q) (6) (ii)] This training is designed for employees who manage waste associated with a Type I or Type I-X Accumulation Point or a Type II or Type II-X Accumulation Area. These employees must be trained to take part in initial response activities, for the purpose of protecting nearby persons, property, or the environment from the effects of a release. They are to be trained in a defensive fashion, without actually trying to stop the release. This course also applies to supervisors over Hazardous Material Processes, Hospital Corpsmen, Occupational Health Nurses, Medical Doctors, Police Officers, Shift Duty Officers, Guard Lieutenants, Police Captains, and Command Duty Officers. Training is to include an 8-hour initial course, and 1-hour annual refresher.

2.4.2.4. Spill Response Technician/Specialist: [29 CFR 1910.120(q)(6)(iii) & (iv)] This training is for Hazardous Waste Handlers, Environmental Protection Specialists, and Fire Department personnel. Training will teach individuals how to respond for the purpose of stopping and mitigating a release of a hazardous substance. Training is to include a 24-hour initial session with an 8-hour annual refresher.

2.4.2.5. Spill Response Incident Commander: [29 CFR 1910.120(q)(6)(v)] This training is designed for employees who assume control of the incident scene, and have the capability to commit resources to the response activities. They may also be

called up to act as the site liaison with Federal, State, local and other government authorities, in regard to clean-up activities this includes the Environmental Protection Supervisor, Environmental Protection Specialists, Fire Chief, Assistant Fire Chiefs, Fire Captains, and Fire Engine Drivers. Training is to include a 24-hour initial session, with an annual 1-hour refresher.

2.5. REPORTING AND RECORDKEEPING

Certain reports and records may be audited by State or Federal inspectors, and therefore must be easily accessible. Records must be carefully preserved and filed where they can be quickly located. The EPD will maintain the following records and reports, as identified in this section and throughout this Plan:

2.5.1. Hazardous Waste Storage Records

2.5.2. Inspection Records

2.5.3. Hazardous Waste Reports

2.5.4. Training Records

2.5.5. Medical Surveillance Program Records

2.5.6. Hazardous Waste Manifests, including:

a. The EPA Uniform Manifest

b. Additional Information Required by Individual States; and

c. Manifest Exception Reporting

2.6. HAZARDOUS WASTE ANALYSIS PLAN AND LAND DISPOSAL RESTRICTION REQUIREMENTS

As an operator of a TSD facility, Crane Division is required, by RCRA, to have a waste analysis plan for Land Disposal Restrictions (LDR), and a land disposal plan. These requirements, along with the reporting and record keeping requirements for the LDR, are found in Appendix 1 of the federal portion of the facility's Final RCRA Part B Permit. This can be provided by the EPD upon request.

2.7. HAZARDOUS WASTE CONTINGENCY PLAN

Crane Division has a separate hazardous waste contingency plan for the facility. The contingency plan is implemented by NAVSURFWARCENDIVINST 5090.5A, Oil and Hazardous Substances Spill Contingency Plan (reference (g)). Note, that reference (g) and subparagraph 1.1.6.5. of this Instruction requires operators, of waste accumulation sites, to prepare site specific contingency plans.

2.8. HAZARDOUS WASTE MINIMIZATION PLAN (HAZMIN PLAN)

Crane Division has a HAZMIN Plan for the facility that is part of reference (k), the NSWC Crane Division Pollution Prevention Plan. The purpose of the HAZMIN Plan is to identify how to reduce the amount of hazardous material used and to reduce the amount of hazardous waste generated. The Pollution Prevention Office (PPO) is responsible for overseeing the implementation of that Plan. Because of its significance, and as required by reference (a), it is cited in this Hazardous Waste Management Plan.

APPENDIX 1

SPECIFIC FEDERAL REGULATIONS, INCLUDING: 40 CFR 261.21-.24 AND 40 CFR 261.31-.33(E)

PLEASE CLICK ON LINK ABOVE TO VIEW REGULATIONS

(i) The nature of the toxicity presented by the constituent.

(ii) The concentration of the constituent in the waste.

(iii) The potential of the constituent or any toxic degradation product of the constituent to migrate from the waste into the environment under the types of improper management considered in paragraph (a)(3)(vii) of this section.

(iv) The persistence of the constituent or any toxic degradation product of the constituent.

(v) The potential for the constituent or any toxic degradation product of the constituent to degrade into non-harmful constituents and the rate of degradation.

(vi) The degree to which the constituent or any degradation product of the constituent bioaccumulates in ecosystems.

(vii) The plausible types of improper management to which the waste could be subjected.

(viii) The quantities of the waste generated at individual generation sites or on a regional or national basis.

(ix) The nature and severity of the human health and environmental damage that has occurred as a result of the improper management of wastes containing the constituent.

(x) Action taken by other governmental agencies or regulatory programs based on the health or environmental hazard posed by the waste or waste constituent.

(xi) Such other factors as may be appropriate.

Substances will be listed on appendix VIII only if they have been shown in scientific studies to have toxic, carcinogenic, mutagenic or teratogenic effects on humans or other life forms.

(Wastes listed in accordance with these criteria will be designated Toxic wastes.)

(b) The Administrator may list classes or types of solid waste as hazardous waste if he has reason to believe that individual wastes, within the class or type of waste, typically or frequently are hazardous under the definition of hazardous waste found in section 1004(5) of the Act.

(c) The Administrator will use the criteria for listing specified in this section to establish the exclusion limits referred to in §261.5(c).

[45 FR 33119, May 19, 1980, as amended at 55 FR 18726, May 4, 1990; 57 FR 14, Jan. 2, 1992]

Subpart C—Characteristics of Hazardous Waste

§261.20 General.

(a) A solid waste, as defined in §261.2, which is not excluded from regulation as a hazardous waste under §261.4(b), is a hazardous waste if it exhibits any of the characteristics identified in this subpart.

[*Comment*: §262.11 of this chapter sets forth the generator's responsibility to determine whether his waste exhibits one or more of the characteristics identified in this subpart]

(b) A hazardous waste which is identified by a characteristic in this subpart is assigned every EPA Hazardous Waste Number that is applicable as set forth in this subpart. This number must be used in complying with the notification requirements of section 3010 of the Act and all applicable recordkeeping and reporting requirements under parts 262 through 265, 268, and 270 of this chapter.

(c) For purposes of this subpart, the Administrator will consider a sample obtained using any of the applicable sampling methods specified in appendix I to be a representative sample within the meaning of part 260 of this chapter.

[*Comment*: Since the appendix I sampling methods are not being formally adopted by the Administrator, a person who desires to employ an alternative sampling method is not required to demonstrate the equivalency of his method under the procedures set forth in §§260.20 and 260.21.]

[45 FR 33119, May 19, 1980, as amended at 51 FR 40636, Nov. 7, 1986; 55 FR 22684, June 1, 1990; 56 FR 3876, Jan. 31, 1991]

§261.21 Characteristic of ignitability.

(a) A solid waste exhibits the characteristic of ignitability if a representative sample of the waste has any of the following properties:

(1) It is a liquid, other than an aqueous solution containing less than 24 percent alcohol by volume and has flash point less than 60°C (140°F), as determined by a Pensky-Martens Closed

Cup Tester, using the test method specified in ASTM Standard D-93-79 or D-93-80 (incorporated by reference, see § 260.11), or a Setaflash Closed Cup Tester, using the test method specified in ASTM Standard D-3278-78 (incorporated by reference, see § 260.11), or as determined by an equivalent test method approved by the Administrator under procedures set forth in §§ 260.20 and 260.21.

(2) It is not a liquid and is capable, under standard temperature and pressure, of causing fire through friction, absorption of moisture or spontaneous chemical changes and, when ignited, burns so vigorously and persistently that it creates a hazard.

(3) It is an ignitable compressed gas as defined in 49 CFR 173.300 and as determined by the test methods described in that regulation or equivalent test methods approved by the Administrator under §§ 260.20 and 260.21.

(4) It is an oxidizer as defined in 49 CFR 173.151.

(b) A solid waste that exhibits the characteristic of ignitability has the EPA Hazardous Waste Number of D001.

[45 FR 33119, May 19, 1980, as amended at 46 FR 35247, July 7, 1981; 55 FR 22684, June 1, 1990]

§ 261.22 Characteristic of corrosivity.

(a) A solid waste exhibits the characteristic of corrosivity if a representative sample of the waste has either of the following properties:

(1) It is aqueous and has a pH less than or equal to 2 or greater than or equal to 12.5, as determined by a pH meter using Method 9040 in "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," EPA Publication SW-846, as incorporated by reference in § 260.11 of this chapter.

(2) It is a liquid and corrodes steel (SAE 1020) at a rate greater than 6.35 mm (0.250 inch) per year at a test temperature of 55°C (130°F) as determined by the test method specified in NACE (National Association of Corrosion Engineers) Standard TM-01-69 as standardized in "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," EPA Publication SW-846, as incorporated by reference in § 260.11 of this chapter.

(b) A solid waste that exhibits the characteristic of corrosivity has the EPA Hazardous Waste Number of D002.

[45 FR 33119, May 19, 1980, as amended at 46 FR 35247, July 7, 1981; 55 FR 22684, June 1, 1990; 58 FR 46049, Aug. 31, 1993]

§ 261.23 Characteristic of reactivity.

(a) A solid waste exhibits the characteristic of reactivity if a representative sample of the waste has *any* of the following properties:

(1) It is normally unstable and readily undergoes violent change without detonating.

(2) It reacts violently with water.

(3) It forms potentially explosive mixtures with water.

(4) When mixed with water, it generates toxic gases, vapors or fumes in a quantity sufficient to present a danger to human health or the environment.

(5) It is a cyanide or sulfide bearing waste which, when exposed to pH conditions between 2 and 12.5, can generate toxic gases, vapors or fumes in a quantity sufficient to present a danger to human health or the environment.

(6) It is capable of detonation or explosive reaction if it is subjected to a strong initiating source or if heated under confinement.

(7) It is readily capable of detonation or explosive decomposition or reaction at standard temperature and pressure.

(8) It is a forbidden explosive as defined in 49 CFR 173.51, or a Class A explosive as defined in 49 CFR 173.53 or a Class B explosive as defined in 49 CFR 173.88.

(b) A solid waste that exhibits the characteristic of reactivity has the EPA Hazardous Waste Number of D003.

[45 FR 33119, May 19, 1980, as amended at 55 FR 22684, June 1, 1990]

§ 261.24 Toxicity characteristic.

(a) A solid waste exhibits the characteristic of toxicity if, using the Toxicity Characteristic Leaching Procedure, test Method 1311 in "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," EPA Publication SW-846, as incorporated by reference in § 260.11 of this chapter, the extract from a representative sample of

the waste contains any of the contaminants listed in table 1 at the concentration equal to or greater than the respective value given in that table. Where the waste contains less than 0.5 percent filterable solids, the waste itself, after filtering using the methodology outlined in Method 1311, is considered to be the extract for the purpose of this section.

(b) A solid waste that exhibits the characteristic of toxicity has the EPA Hazardous Waste Number specified in Table I which corresponds to the toxic contaminant causing it to be hazardous.

TABLE 1—MAXIMUM CONCENTRATION OF CONTAMINANTS FOR THE TOXICITY CHARACTERISTIC

EPA HW No.[1]	Contaminant	CAS No.[2]	Regulatory Level (mg/L)
D004	Arsenic	7440-38-2	5.0
D005	Barium	7440-39-3	100.0
D018	Benzene	71-43-2	0.5
D006	Cadmium	7440-43-9	1.0
D019	Carbon tetrachloride	56-23-5	0.5
D020	Chlordane	57-74-9	0.03
D021	Chlorobenzene	108-90-7	100.0
D022	Chloroform	67-66-3	6.0
D007	Chromium	7440-47-3	5.0
D023	o-Cresol	95-48-7	[4]200.0
D024	m-Cresol	108-39-4	[4]200.0
D025	p-Cresol	106-44-5	[4]200.0
D026	Cresol		[4]200.0
D016	2,4-D	94-75-7	10.0
D027	1,4-Dichlorobenzene	106-46-7	7.5
D028	1,2-Dichloroethane	107-06-2	0.5
D029	1,1-Dichloroethylene	75-35-4	0.7
D030	2,4-Dinitrotoluene	121-14-2	[3]0.13
D012	Endrin	72-20-8	0.02
D031	Heptachlor (and its epoxide).	76-44-8	0.008
D032	Hexachlorobenzene	118-74-1	[3]0.13
D033	Hexachlorobutadiene	87-68-3	0.5
D034	Hexachloroethane	67-72-1	3.0
D008	Lead	7439-92-1	5.0
D013	Lindane	58-89-9	0.4
D009	Mercury	7439-97-6	0.2
D014	Methoxychlor	72-43-5	10.0
D035	Methyl ethyl ketone	78-93-3	200.0
D036	Nitrobenzene	98-95-3	2.0
D037	Pentrachlorophenol	87-86-5	100.0
D038	Pyridine	110-86-1	[3]5.0
D010	Selenium	7782-49-2	1.0
D011	Silver	7440-22-4	5.0
D039	Tetrachloroethylene	127-18-4	0.7
D015	Toxaphene	8001-35-2	0.5
D040	Trichloroethylene	79-01-6	0.5
D041	2,4,5-Trichlorophenol	95-95-4	400.0
D042	2,4,6-Trichlorophenol	88-06-2	2.0
D017	2,4,5-TP (Silvex)	93-72-1	1.0
D043	Vinyl chloride	75-01-4	0.2

[1] Hazardous waste number.

[2] Chemical abstracts service number.

[3] Quantitation limit is greater than the calculated regulatory level. The quantitation limit therefore becomes the regulatory level.

[4] If o-, m-, and p-Cresol concentrations cannot be differentiated, the total cresol (D026) concentration is used. The regulatory level of total cresol is 200 mg/l.

[55 FR 11862, Mar. 29, 1990, as amended at 55 FR 22684, June 1, 1990; 55 FR 26987, June 29, 1990; 58 FR 46049, Aug. 31, 1993]

Subpart D—Lists of Hazardous Wastes

§261.30 General.

(a) A solid waste is a hazardous waste if it is listed in this subpart, unless it has been excluded from this list under §§260.20 and 260.22.

(b) The Administrator will indicate his basis for listing the classes or types of wastes listed in this subpart by employing one or more of the following Hazard Codes:

Ignitable Waste	(I)
Corrosive Waste	(C)
Reactive Waste	(R)
Toxicity Characteristic Waste	(E)
Acute Hazardous Waste	(H)
Toxic Waste	(T)

Appendix VII identifies the constituent which caused the Administrator to list the waste as a Toxicity Characteristic Waste (E) or Toxic Waste (T) in §§261.31 and 261.32.

(c) Each hazardous waste listed in this subpart is assigned an EPA Hazardous Waste Number which precedes the name of the waste. This number must be used in complying with the notification requirements of Section 3010 of the Act and certain recordkeeping and reporting requirements under parts 262 through 265, 268, and part 270 of this chapter.

(d) The following hazardous wastes listed in §261.31 or §261.32 are subject to the exclusion limits for acutely hazardous wastes established in §261.5: EPA Hazardous Wastes Nos. F020, F021, F022, F023, F026, and F027.

[45 FR 33119, May 19, 1980, as amended at 48 FR 14294, Apr. 1, 1983; 50 FR 2000, Jan. 14, 1985; 51 FR 40636, Nov. 7, 1986; 55 FR 11863, Mar. 29, 1990]

§ 261.31 Hazardous wastes from non-specific sources.

(a) The following solid wastes are listed hazardous wastes from non-specific sources unless they are excluded under §§ 260.20 and 260.22 and listed in appendix IX.

Industry and EPA hazardous waste No.	Hazardous waste	Hazard code
Generic:		
F001	The following spent halogenated solvents used in degreasing: Tetrachloroethylene, trichloroethylene, methylene chloride, 1,1,1-trichloroethane, carbon tetrachloride, and chlorinated fluorocarbons; all spent solvent mixtures/blends used in degreasing containing, before use, a total of ten percent or more (by volume) of one or more of the above halogenated solvents or those solvents listed in F002, F004, and F005; and still bottoms from the recovery of these spent solvents and spent solvent mixtures.	(T)
F002	The following spent halogenated solvents: Tetrachloroethylene, methylene chloride, trichloroethylene, 1,1,1-trichloroethane, chlorobenzene, 1,1,2-trichloro-1,2,2-trifluoroethane, ortho-dichlorobenzene, trichlorofluoromethane, and 1,1,2-trichloroethane; all spent solvent mixtures/blends containing, before use, a total of ten percent or more (by volume) of one or more of the above halogenated solvents or those listed in F001, F004, or F005; and still bottoms from the recovery of these spent solvents and spent solvent mixtures.	(T)
F003	The following spent non-halogenated solvents: Xylene, acetone, ethyl acetate, ethyl benzene, ethyl ether, methyl isobutyl ketone, n-butyl alcohol, cyclohexanone, and methanol; all spent solvent mixtures/blends containing, before use, only the above spent non-halogenated solvents; and all spent solvent mixtures/blends containing, before use, one or more of the above non-halogenated solvents, and, a total of ten percent or more (by volume) of one or more of those solvents listed in F001, F002, F004, and F005; and still bottoms from the recovery of these spent solvents and spent solvent mixtures.	(I)*
F004	The following spent non-halogenated solvents: Cresols and cresylic acid, and nitrobenzene; all spent solvent mixtures/blends containing, before use, a total of ten percent or more (by volume) of one or more of the above non-halogenated solvents or those solvents listed in F001, F002, and F005; and still bottoms from the recovery of these spent solvents and spent solvent mixtures.	(T)
F005	The following spent non-halogenated solvents: Toluene, methyl ethyl ketone, carbon disulfide, isobutanol, pyridine, benzene, 2-ethoxyethanol, and 2-nitropropane; all spent solvent mixtures/blends containing, before use, a total of ten percent or more (by volume) of one or more of the above non-halogenated solvents or those solvents listed in F001, F002, or F004; and still bottoms from the recovery of these spent solvents and spent solvent mixtures.	(I,T)
F006	Wastewater treatment sludges from electroplating operations except from the following processes: (1) Sulfuric acid anodizing of aluminum; (2) tin plating on carbon steel; (3) zinc plating (segregated basis) on carbon steel; (4) aluminum or zinc-aluminum plating on carbon steel; (5) cleaning/stripping associated with tin, zinc and aluminum plating on carbon steel; and (6) chemical etching and milling of aluminum.	(T)
F007	Spent cyanide plating bath solutions from electroplating operations	(R, T)
F008	Plating bath residues from the bottom of plating baths from electroplating operations where cyanides are used in the process.	(R, T)
F009	Spent stripping and cleaning bath solutions from electroplating operations where cyanides are used in the process.	(R, T)
F010	Quenching bath residues from oil baths from metal heat treating operations where cyanides are used in the process.	(R, T)
F011	Spent cyanide solutions from salt bath pot cleaning from metal heat treating operations.	(R, T)
F012	Quenching waste water treatment sludges from metal heat treating operations where cyanides are used in the process.	(T)
F019	Wastewater treatment sludges from the chemical conversion coating of aluminum except from zirconium phosphating in aluminum can washing when such phosphating is an exclusive conversion coating process.	(T)
F020	Wastes (except wastewater and spent carbon from hydrogen chloride purification) from the production or manufacturing use (as a reactant, chemical intermediate, or component in a formulating process) of tri- or tetrachlorophenol, or of intermediates used to produce their pesticide derivatives. (This listing does not include wastes from the production of Hexachlorophene from highly purified 2,4,5-trichlorophenol.).	(H)
F021	Wastes (except wastewater and spent carbon from hydrogen chloride purification) from the production or manufacturing use (as a reactant, chemical intermediate, or component in a formulating process) of pentachlorophenol, or of intermediates used to produce its derivatives.	(H)
F022	Wastes (except wastewater and spent carbon from hydrogen chloride purification) from the manufacturing use (as a reactant, chemical intermediate, or component in a formulating process) of tetra-, penta-, or hexachlorobenzenes under alkaline conditions	(H)

Industry and EPA hazardous waste No.	Hazardous waste	Hazard code
F023	Wastes (except wastewater and spent carbon from hydrogen chloride purification) from the production of materials on equipment previously used for the production or manufacturing use (as a reactant, chemical intermediate, or component in a formulating process) of tri- and tetrachlorophenols. (This listing does not include wastes from equipment used only for the production or use of Hexachlorophene from highly purified 2,4,5-trichlorophenol.).	(H)
F024	Process wastes, including but not limited to, distillation residues, heavy ends, tars, and reactor clean-out wastes, from the production of certain chlorinated aliphatic hydrocarbons by free radical catalyzed processes. These chlorinated aliphatic hydrocarbons are those having carbon chain lengths ranging from one to and including five, with varying amounts and positions of chlorine substitution. (This listing does not include wastewaters, wastewater treatment sludges, spent catalysts, and wastes listed in §261.31 or §261.32.).	(T)
F025	Condensed light ends, spent filters and filter aids, and spent desiccant wastes from the production of certain chlorinated aliphatic hydrocarbons, by free radical catalyzed processes. These chlorinated aliphatic hydrocarbons are those having carbon chain lengths ranging from one to and including five, with varying amounts and positions of chlorine substitution.	(T)
F026	Wastes (except wastewater and spent carbon from hydrogen chloride purification) from the production of materials on equipment previously used for the manufacturing use (as a reactant, chemical intermediate, or component in a formulating process) of tetra-, penta-, or hexachlorobenzene under alkaline conditions.	(H)
F027	Discarded unused formulations containing tri-, tetra-, or pentachlorophenol or discarded unused formulations containing compounds derived from these chlorophenols. (This listing does not include formulations containing Hexachlorophene sythesized from prepurified 2,4,5-trichlorophenol as the sole component.).	(H)
F028	Residues resulting from the incineration or thermal treatment of soil contaminated with EPA Hazardous Waste Nos. F020, F021, F022, F023, F026, and F027.	(T)
F032	Wastewaters (except those that have not come into contact with process contaminants), process residuals, preservative drippage, and spent formulations from wood preserving processes generated at plants that currently use or have previously used chlorophenolic formulations (except potentially cross-contaminated wastes that have had the F032 waste code deleted in accordance with §261.35 of this chapter or potentially cross-contaminated wastes that are otherwise currently regulated as hazardous wastes (i.e., F034 or F035), and where the generator does not resume or initiate use of chlorophenolic formulations). This listing does not include K001 bottom sediment sludge from the treatment of wastewater from wood preserving processes that use creosote and/or pentachlorophenol.	(T)
F034	Wastewaters (except those that have not come into contact with process contaminants), process residuals, preservative drippage, and spent formulations from wood preserving processes generated at plants that use creosote formulations. This listing does not include K001 bottom sediment sludge from the treatment of wastewater from wood preserving processes that use creosote and/or pentachlorophenol.	(T)
F035	Wastewaters (except those that have not come into contact with process contaminants), process residuals, preservative drippage, and spent formulations from wood preserving processes generated at plants that use inorganic preservatives containing arsenic or chromium. This listing does not include K001 bottom sediment sludge from the treatment of wastewater from wood preserving processes that use creosote and/or pentachlorophenol.	(T)
F037	Petroleum refinery primary oil/water/solids separation sludge—Any sludge generated from the gravitational separation of oil/water/solids during the storage or treatment of process wastewaters and oily cooling wastewaters from petroleum refineries. Such sludges include, but are not limited to, those generated in: oil/water/solids separators; tanks and impoundments; ditches and other conveyances; sumps; and stormwater units receiving dry weather flow. Sludge generated in stormwater units that do not receive dry weather flow, sludges generated from non-contact once-through cooling waters segregated for treatment from other process or oily cooling waters, sludges generated in aggressive biological treatment units as defined in §261.31(b)(2) (including sludges generated in one or more additional units after wastewaters have been treated in aggressive biological treatment units) and K051 wastes are not included in this listing.	(T)

Industry and EPA hazardous waste No.	Hazardous waste	Hazard code
F038	Petroleum refinery secondary (emulsified) oil/water/solids separation sludge—Any sludge and/or float generated from the physical and/or chemical separation of oil/water/solids in process wastewaters and oily cooling wastewaters from petroleum refineries. Such wastes include, but are not limited to, all sludges and floats generated in: induced air flotation (IAF) units, tanks and impoundments, and all sludges generated in DAF units. Sludges generated in stormwater units that do not receive dry weather flow, sludges generated from non-contact once-through cooling waters segregated for treatment from other process or oily cooling waters, sludges and floats generated in aggressive biological treatment units as defined in § 261.31(b)(2) (including sludges and floats generated in one or more additional units after wastewaters have been treated in aggressive biological treatment units) and F037, K048, and K051 wastes are not included in this listing.	(T)
F039	Leachate (liquids that have percolated through land disposed wastes) resulting from the disposal of more than one restricted waste classified as hazardous under subpart D of this part. (Leachate resulting from the disposal of one or more of the following EPA Hazardous Wastes and no other Hazardous Wastes retains its EPA Hazardous Waste Number(s): F020, F021, F022, F026, F027, and/or F028.).	(T)

(b) Listing Specific Definitions: (1) For the purposes of the F037 and F038 listings, oil/water/solids is defined as oil and/or water and/or solids.

(2) (i) For the purposes of the F037 and F038 listings, aggressive biological treatment units are defined as units which employ one of the following four treatment methods: activated sludge; trickling filter; rotating biological contactor for the continuous accelerated biological oxidation of wastewaters; or high-rate aeration. High-rate aeration is a system of surface impoundments or tanks, in which intense mechanical aeration is used to completely mix the wastes, enhance biological activity, and (A) the units employ a minimum of 6 hp per million gallons of treatment volume; and either (B) the hydraulic retention time of the unit is no longer than 5 days; or (C) the hydraulic retention time is no longer than 30 days and the unit does not generate a sludge that is a hazardous waste by the Toxicity Characteristic.

(ii) Generators and treatment, storage and disposal facilities have the burden of proving that their sludges are exempt from listing as F037 and F038 wastes under this definition. Generators and treatment, storage and disposal facilities must maintain, in their operating or other onsite records, documents and data sufficient to prove that: (A) the unit is an aggressive biological treatment unit as defined in this subsection; and (B) the sludges sought to be exempted from the definitions of F037 and/or F038 were actually generated in the aggressive biological treatment unit.

(3) (i) For the purposes of the F037 listing, sludges are considered to be generated at the moment of deposition in the unit, where deposition is defined as at least a temporary cessation of lateral particle movement.

(ii) For the purposes of the F038 listing,

(A) sludges are considered to be generated at the moment of deposition in the unit, where deposition is defined as at least a temporary cessation of lateral particle movement and

(B) floats are considered to be generated at the moment they are formed in the top of the unit.

[46 FR 4617, Jan. 16, 1981, as amended at 60 FR 33913, June 29, 1995]

EDITORIAL NOTE: For FEDERAL REGISTER citations affecting § 261.31, see the List of CFR Sections Affected in the Finding Aids section of this volume.

§ 261.32 Hazardous wastes from specific sources.

The following solid wastes are listed hazardous wastes from specific sources unless they are excluded under §§ 260.20 and 260.22 and listed in appendix IX.

Industry and EPA hazardous waste No.	Hazardous waste	Hazard code
Wood preservation: K001	Bottom sediment sludge from the treatment of wastewaters from wood preserving processes that use creosote and/or pentachlorophenol	(T)

Industry and EPA hazardous waste No.	Hazardous waste	Hazard code
Inorganic pigments:		
K002	Wastewater treatment sludge from the production of chrome yellow and orange pigments.	(T)
K003	Wastewater treatment sludge from the production of molybdate orange pigments	(T)
K004	Wastewater treatment sludge from the production of zinc yellow pigments	(T)
K005	Wastewater treatment sludge from the production of chrome green pigments	(T)
K006	Wastewater treatment sludge from the production of chrome oxide green pigments (anhydrous and hydrated).	(T)
K007	Wastewater treatment sludge from the production of iron blue pigments	(T)
K008	Oven residue from the production of chrome oxide green pigments	(T)
Organic chemicals:		
K009	Distillation bottoms from the production of acetaldehyde from ethylene	(T)
K010	Distillation side cuts from the production of acetaldehyde from ethylene	(T)
K011	Bottom stream from the wastewater stripper in the production of acrylonitrile	(R, T)
K013	Bottom stream from the acetonitrile column in the production of acrylonitrile	(R, T)
K014	Bottoms from the acetonitrile purification column in the production of acrylonitrile	(T)
K015	Still bottoms from the distillation of benzyl chloride	(T)
K016	Heavy ends or distillation residues from the production of carbon tetrachloride	(T)
K017	Heavy ends (still bottoms) from the purification column in the production of epichlorohydrin.	(T)
K018	Heavy ends from the fractionation column in ethyl chloride production	(T)
K019	Heavy ends from the distillation of ethylene dichloride in ethylene dichloride production.	(T)
K020	Heavy ends from the distillation of vinyl chloride in vinyl chloride monomer production	(T)
K021	Aqueous spent antimony catalyst waste from fluoromethanes production	(T)
K022	Distillation bottom tars from the production of phenol/acetone from cumene	(T)
K023	Distillation light ends from the production of phthalic anhydride from naphthalene	(T)
K024	Distillation bottoms from the production of phthalic anhydride from naphthalene	(T)
K025	Distillation bottoms from the production of nitrobenzene by the nitration of benzene	(T)
K026	Stripping still tails from the production of methy ethyl pyridines	(T)
K027	Centrifuge and distillation residues from toluene diisocyanate production	(R, T)
K028	Spent catalyst from the hydrochlorinator reactor in the production of 1,1,1-trichloroethane.	(T)
K029	Waste from the product steam stripper in the production of 1,1,1-trichloroethane	(T)
K030	Column bottoms or heavy ends from the combined production of trichloroethylene and perchloroethylene.	(T)
K083	Distillation bottoms from aniline production	(T)
K085	Distillation or fractionation column bottoms from the production of chlorobenzenes	(T)
K093	Distillation light ends from the production of phthalic anhydride from ortho-xylene	(T)
K094	Distillation bottoms from the production of phthalic anhydride from ortho-xylene	(T)
K095	Distillation bottoms from the production of 1,1,1-trichloroethane	(T)
K096	Heavy ends from the heavy ends column from the production of 1,1,1-trichloroethane	(T)
K103	Process residues from aniline extraction from the production of aniline	(T)
K104	Combined wastewater streams generated from nitrobenzene/aniline production	(T)
K105	Separated aqueous stream from the reactor product washing step in the production of chlorobenzenes.	(T)
K107	Column bottoms from product separation from the production of 1,1-dimethyl-hydrazine (UDMH) from carboxylic acid hydrazines.	(C,T)
K108	Condensed column overheads from product separation and condensed reactor vent gases from the production of 1,1-dimethylhydrazine (UDMH) from carboxylic acid hydrazides.	(I,T)
K109	Spent filter cartridges from product purification from the production of 1,1-dimethylhydrazine (UDMH) from carboxylic acid hydrazides.	(T)
K110	Condensed column overheads from intermediate separation from the production of 1,1-dimethylhydrazine (UDMH) from carboxylic acid hydrazides.	(T)
K111	Product washwaters from the production of dinitrotoluene via nitration of toluene	(C,T)
K112	Reaction by-product water from the drying column in the production of toluenediamine via hydrogenation of dinitrotoluene.	(T)
K113	Condensed liquid light ends from the purification of toluenediamine in the production of toluenediamine via hydrogenation of dinitrotoluene.	(T)
K114	Vicinals from the purification of toluenediamine in the production of toluenediamine via hydrogenation of dinitrotoluene.	(T)
K115	Heavy ends from the purification of toluenediamine in the production of toluenediamine via hydrogenation of dinitrotoluene.	(T)
K116	Organic condensate from the solvent recovery column in the production of toluene diisocyanate via phosgenation of toluenediamine.	(T)
K117	Wastewater from the reactor vent gas scrubber in the production of ethylene dibromide via bromination of ethene.	(T)
K118	Spent adsorbent solids from purification of ethylene dibromide in the production of ethylene dibromide via bromination of ethene.	(T)
K136	Still bottoms from the purification of ethylene dibromide in the production of ethylene dibromide via bromination of ethene.	(T)

Industry and EPA hazardous waste No.	Hazardous waste	Hazard code
K149	Distillation bottoms from the production of alpha- (or methyl-) chlorinated toluenes, ring-chlorinated toluenes, benzoyl chlorides, and compounds with mixtures of these functional groups. (This waste does not include still bottoms from the distillation of benzyl chloride.).	(T)
K150	Organic residuals, excluding spent carbon adsorbent, from the spent chlorine gas and hydrochloric acid recovery processes associated with the production of alpha- (or methyl-) chlorinated toluenes, ring-chlorinated toluenes, benzoyl chlorides, and compounds with mixtures of these functional groups.	(T)
K151	Wastewater treatment sludges, excluding neutralization and biological sludges, generated during the treatment of wastewaters from the production of alpha- (or methyl-) chlorinated toluenes, ring-chlorinated toluenes, benzoyl chlorides, and compounds with mixtures of these functional groups.	(T)
K156	Organic waste (including heavy ends, still bottoms, light ends, spent solvents, filtrates, and decantates) from the production of carbamates and carbamoyl oximes. (This listing does not apply to wastes generated from the manufacture of 3-iodo-2-propynyl n-butylcarbamate.).	(T)
K157	Wastewaters (including scrubber waters, condenser waters, washwaters, and separation waters) from the production of carbamates and carbamoyl oximes. (This listing does not apply to wastes generated from the manufacture of 3-iodo-2-propynyl n-butylcarbamate.).	(T)
K158	Bag house dusts and filter/separation solids from the production of carbamates and carbamoyl oximes. (This listing does not apply to wastes generated from the manufacture of 3-iodo-2-propynyl n-butylcarbamate.).	(T)
K159	Organics from the treatment of thiocarbamate wastes	(T)
K161	Purification solids (including filtration, evaporation, and centrifugation solids), bag house dust and floor sweepings from the production of dithiocarbamate acids and their salts. (This listing does not include K125 or K126.).	(R,T)
Inorganic chemicals:		
K071	Brine purification muds from the mercury cell process in chlorine production, where separately prepurified brine is not used.	(T)
K073	Chlorinated hydrocarbon waste from the purification step of the diaphragm cell process using graphite anodes in chlorine production.	(T)
K106	Wastewater treatment sludge from the mercury cell process in chlorine production	(T)
Pesticides:		
K031	By-product salts generated in the production of MSMA and cacodylic acid	(T)
K032	Wastewater treatment sludge from the production of chlordane	(T)
K033	Wastewater and scrub water from the chlorination of cyclopentadiene in the production of chlordane.	(T)
K034	Filter solids from the filtration of hexachlorocyclopentadiene in the production of chlordane.	(T)
K035	Wastewater treatment sludges generated in the production of creosote	(T)
K036	Still bottoms from toluene reclamation distillation in the production of disulfoton	(T)
K037	Wastewater treatment sludges from the production of disulfoton	(T)
K038	Wastewater from the washing and stripping of phorate production	(T)
K039	Filter cake from the filtration of diethylphosphorodithioic acid in the production of phorate.	(T)
K040	Wastewater treatment sludge from the production of phorate	(T)
K041	Wastewater treatment sludge from the production of toxaphene	(T)
K042	Heavy ends or distillation residues from the distillation of tetrachlorobenzene in the production of 2,4,5-T.	(T)
K043	2,6-Dichlorophenol waste from the production of 2,4-D	(T)
K097	Vacuum stripper discharge from the chlordane chlorinator in the production of chlordane.	(T)
K098	Untreated process wastewater from the production of toxaphene	(T)
K099	Untreated wastewater from the production of 2,4-D	(T)
K123	Process wastewater (including supernates, filtrates, and washwaters) from the production of ethylenebisdithiocarbamic acid and its salt.	(T)
K124	Reactor vent scrubber water from the production of ethylenebisdithiocarbamic acid and its salts.	(C, T)
K125	Filtration, evaporation, and centrifugation solids from the production of ethylenebisdithiocarbamic acid and its salts.	(T)
K126	Baghouse dust and floor sweepings in milling and packaging operations from the production or formulation of ethylenebisdithiocarbamic acid and its salts.	(T)
K131	Wastewater from the reactor and spent sulfuric acid from the acid dryer from the production of methyl bromide.	(C, T)
K132	Spent absorbent and wastewater separator solids from the production of methyl bromide.	(T)
Explosives:		
K044	Wastewater treatment sludges from the manufacturing and processing of explosives	(R)
K045	Spent carbon from the treatment of wastewater containing explosives	(R)
K046	Wastewater treatment sludges from the manufacturing, formulation and loading of lead-based initiating compounds.	(T)
K047	Pink/red water from TNT operations	(R)

Industry and EPA hazardous waste No.	Hazardous waste	Hazard code
Petroleum refining:		
K048	Dissolved air flotation (DAF) float from the petroleum refining industry	(T)
K049	Slop oil emulsion solids from the petroleum refining industry	(T)
K050	Heat exchanger bundle cleaning sludge from the petroleum refining industry	(T)
K051	API separator sludge from the petroleum refining industry	(T)
K052	Tank bottoms (leaded) from the petroleum refining industry	(T)
Iron and steel:		
K061	Emission control dust/sludge from the primary production of steel in electric furnaces	(T)
K062	Spent pickle liquor generated by steel finishing operations of facilities within the iron and steel industry (SIC Codes 331 and 332).	(C,T)
Primary copper:		
K064	Acid plant blowdown slurry/sludge resulting from the thickening of blowdown slurry from primary copper production.	(T)
Primary lead:		
K065	Surface impoundment solids contained in and dredged from surface impoundments at primary lead smelting facilities.	(T)
Primary zinc:		
K066	Sludge from treatment of process wastewater and/or acid plant blowdown from primary zinc production.	(T)
Primary aluminum:		
K088	Spent potliners from primary aluminum reduction	(T)
Ferroalloys:		
K090	Emission control dust or sludge from ferrochromiumsilicon production	(T)
K091	Emission control dust or sludge from ferrochromium production	(T)
Secondary lead:		
K069	Emission control dust/sludge from secondary lead smelting. (NOTE: This listing is stayed administratively for sludge generated from secondary acid scrubber systems. The stay will remain in effect until further administrative action is taken. If EPA takes further action effecting this stay, EPA will publish a notice of the action in the Federal Register.	(T)
K100	Waste leaching solution from acid leaching of emission control dust/sludge from secondary lead smelting.	(T)
Veterinary pharmaceuticals:		
K084	Wastewater treatment sludges generated during the production of veterinary pharmaceuticals from arsenic or organo-arsenic compounds.	(T)
K101	Distillation tar residues from the distillation of aniline-based compounds in the production of veterinary pharmaceuticals from arsenic or organo-arsenic compounds.	(T)
K102	Residue from the use of activated carbon for decolorization in the production of veterinary pharmaceuticals from arsenic or organo-arsenic compounds.	(T)
Ink formulation:		
K086	Solvent washes and sludges, caustic washes and sludges, or water washes and sludges from cleaning tubs and equipment used in the formulation of ink from pigments, driers, soaps, and stabilizers containing chromium and lead.	(T)
Coking:		
K060	Ammonia still lime sludge from coking operations	(T)
K087	Decanter tank tar sludge from coking operations	(T)
K141	Process residues from the recovery of coal tar, including, but not limited to, collecting sump residues from the production of coke from coal or the recovery of coke by-products produced from coal. This listing does not include K087 (decanter tank tar sludges from coking operations).	(T)
K142	Tar storage tank residues from the production of coke from coal or from the recovery of coke by-products produced from coal.	(T)
K143	Process residues from the recovery of light oil, including, but not limited to, those generated in stills, decanters, and wash oil recovery units from the recovery of coke by-products produced from coal.	(T)
K144	Wastewater sump residues from light oil refining, including, but not limited to, intercepting or contamination sump sludges from the recovery of coke by-products produced from coal.	(T)
K145	Residues from naphthalene collection and recovery operations from the recovery of coke by-products produced from coal.	(T)
K147	Tar storage tank residues from coal tar refining	(T)
K148	Residues from coal tar distillation, including but not limited to, still bottoms	(T)

[46 FR 4618, Jan. 16, 1981]

EDITORIAL NOTE: For FEDERAL REGISTER citations affecting § 261.32, see the List of CFR Sections Affected in the Finding Aids section of this volume.

§ 261.33 Discarded commercial chemical products, off-specification species, container residues, and spill residues thereof.

The following materials or items are hazardous wastes if and when they are discarded or intended to be discarded as described in § 261.2(a)(2)(i), when they are mixed with waste oil or used oil or other material and applied to the land for dust suppression or road treatment, when they are otherwise applied to the land in lieu of their original intended use or when they are contained in products that are applied to the land in lieu of their original intended use, or when, in lieu of their original intended use, they are produced for use as (or as a component of) a fuel, distributed for use as a fuel, or burned as a fuel.

(a) Any commercial chemical product, or manufacturing chemical intermediate having the generic name listed in paragraph (e) or (f) of this section.

(b) Any off-specification commercial chemical product or manufacturing chemical intermediate which, if it met specifications, would have the generic name listed in paragraph (e) or (f) of this section.

(c) Any residue remaining in a container or in an inner liner removed from a container that has held any commercial chemical product or manufacturing chemical intermediate having the generic name listed in paragraphs (e) or (f) of this section, unless the container is empty as defined in § 261.7(b) of this chapter.

[*Comment:* Unless the residue is being beneficially used or reused, or legitimately recycled or reclaimed; or being accumulated, stored, transported or treated prior to such use, re-use, recycling or reclamation, EPA considers the residue to be intended for discard, and thus, a hazardous waste. An example of a legitimate re-use of the residue would be where the residue remains in the container and the container is used to hold the same commercial chemical product or manufacturing chemical intermediate it previously held. An example of the discard of the residue would be where the drum is sent to a drum reconditioner who reconditions the drum but discards the residue.]

(d) Any residue or contaminated soil, water or other debris resulting from the cleanup of a spill into or on any land or water of any commercial chemical product or manufacturing chemical intermediate having the generic name listed in paragraph (e) or (f) of this section, or any residue or contaminated soil, water or other debris resulting from the cleanup of a spill, into or on any land or water, of any off-specification chemical product and manufacturing chemical intermediate which, if it met specifications, would have the generic name listed in paragraph (e) or (f) of this section.

[*Comment:* The phrase "commercial chemical product or manufacturing chemical intermediate having the generic name listed in . . ." refers to a chemical substance which is manufactured or formulated for commercial or manufacturing use which consists of the commercially pure grade of the chemical, any technical grades of the chemical that are produced or marketed, and all formulations in which the chemical is the sole active ingredient. It does not refer to a material, such as a manufacturing process waste, that contains any of the substances listed in paragraph (e) or (f). Where a manufacturing process waste is deemed to be a hazardous waste because it contains a substance listed in paragraph (e) or (f), such waste will be listed in either § 261.31 or § 261.32 or will be identified as a hazardous waste by the characteristics set forth in subpart C of this part.]

(e) The commercial chemical products, manufacturing chemical intermediates or off-specification commercial chemical products or manufacturing chemical intermediates referred to in paragraphs (a) through (d) of this section, are identified as acute hazardous wastes (H) and are subject to be the small quantity exclusion defined in § 261.5(e).

[*Comment:* For the convenience of the regulated community the primary hazardous properties of these materials have been indicated by the letters T (Toxicity), and R (Reactivity). Absence of a letter indicates that the compound only is listed for acute toxicity.]

These wastes and their corresponding EPA Hazardous Waste Numbers are:

Hazardous waste No.	Chemical abstracts No.	Substance
P023	107-20-0	Acetaldehyde, chloro-
P002	591-08-2	Acetamide, N-(aminothioxomethyl)-
P057	640-19-7	Acetamide, 2-fluoro-
P058	62-74-8	Acetic acid, fluoro-, sodium salt
P002	591-08-2	1-Acetyl-2-thiourea
P003	107-02-8	Acrolein.
P070	116-06-3	Aldicarb
P203	1646-88-4	Aldicarb sulfone.
P004	309-00-2	Aldrin
P005	107-18-6	Allyl alcohol
P006	20859-73-8	Aluminum phosphide (R,T)
P007	2763-96-4	5-(Aminomethyl)-3-isoxazolol
P008	504-24-5	4-Aminopyridine
P009	131-74-8	Ammonium picrate (R)
P119	7803-55-6	Ammonium vanadate
P099	506-61-6	Argentate(1-), bis(cyano-C)-, potassium
P010	7778-39-4	Arsenic acid H_3AsO_4
P012	1327-53-3	Arsenic oxide As_2O_3
P011	1303-28-2	Arsenic oxide As_2O_5
P011	1303-28-2	Arsenic pentoxide
P012	1327-53-3	Arsenic trioxide
P038	692-42-2	Arsine, diethyl-
P036	696-28-6	Arsonous dichloride, phenyl-
P054	151-56-4	Aziridine
P067	75-55-8	Aziridine, 2-methyl-
P013	542-62-1	Barium cyanide
P024	106-47-8	Benzenamine, 4-chloro-
P077	100-01-6	Benzenamine, 4-nitro-
P028	100-44-7	Benzene, (chloromethyl)-
P042	51-43-4	1,2-Benzenediol, 4-[1-hydroxy-2-(methylamino)ethyl]-, (R)-
P046	122-09-8	Benzeneethanamine, alpha,alpha-dimethyl-
P014	108-98-5	Benzenethiol
P127	1563-66-2	7-Benzofuranol, 2,3-dihydro-2,2-dimethyl-, methylcarbamate.
P188	57-64-7	Benzoic acid, 2-hydroxy-, compd. with (3aS-cis)-1,2,3,3a,8,8a-hexahydro-1,3a,8-trimethylpyrrolo[2,3-b]indol-5-yl methylcarbamate ester (1:1).
P001	181-81-2	2H-1-Benzopyran-2-one, 4-hydroxy-3-(3-oxo-1-phenylbutyl)-, & salts, when present at concentrations greater than 0.3%
P028	100-44-7	Benzyl chloride
P015	7440-41-7	Beryllium powder
P017	598-31-2	Bromoacetone
P018	357-57-3	Brucine
P045	39196-18-4	2-Butanone, 3,3-dimethyl-1-(methylthio)-, O-[methylamino)carbonyl] oxime
P021	592-01-8	Calcium cyanide
P021	592-01-8	Calcium cyanide $Ca(CN)_2$
P189	55285-14-8	Carbamic acid, [(dibutylamino)- thio]methyl-, 2,3-dihydro-2,2-dimethyl- 7-benzofuranyl ester.
P191	644-64-4	Carbamic acid, dimethyl-, 1-[(dimethyl-amino)carbonyl]- 5-methyl-1H- pyrazol-3-yl ester.
P192	119-38-0	Carbamic acid, dimethyl-, 3-methyl-1- (1-methylethyl)-1H- pyrazol-5-yl ester.
P190	1129-41-5	Carbamic acid, methyl-, 3-methylphenyl ester.
P127	1563-66-2	Carbofuran.
P022	75-15-0	Carbon disulfide
P095	75-44-5	Carbonic dichloride
P189	55285-14-8	Carbosulfan.
P023	107-20-0	Chloroacetaldehyde
P024	106-47-8	p-Chloroaniline
P026	5344-82-1	1-(o-Chlorophenyl)thiourea
P027	542-76-7	3-Chloropropionitrile
P029	544-92-3	Copper cyanide
P029	544-92-3	Copper cyanide Cu(CN)
P202	64-00-6	m-Cumenyl methylcarbamate.
P030	.....	Cyanides (soluble cyanide salts), not otherwise specified
P031	460-19-5	Cyanogen
P033	506-77-4	Cyanogen chloride
P033	506-77-4	Cyanogen chloride (CN)Cl
P034	131-89-5	2-Cyclohexyl-4,6-dinitrophenol
P016	542-88-1	Dichloromethyl ether
P036	696-28-6	Dichlorophenylarsine
P037	60-57-1	Dieldrin
P038	692-42-2	Diethylarsine
P041	311-45-5	Diethyl-p-nitrophenyl phosphate
P040	297-97-2	O,O-Diethyl O-pyrazinyl phosphorothioate
P043	55-91-4	Diisopropylfluorophosphate (DFP)

Hazardous waste No.	Chemical abstracts No.	Substance
P004	309–00–2	1,4,5,8-Dimethanonaphthalene, 1,2,3,4,10,10-hexa- chloro-1,4,4a,5,8,8a,-hexahydro-, (1alpha,4alpha,4abeta,5alpha,8alpha,8abeta)-
P060	465–73–6	1,4,5,8-Dimethanonaphthalene, 1,2,3,4,10,10-hexa- chloro-1,4,4a,5,8,8a-hexahydro-, (1alpha,4alpha,4abeta,5beta,8beta,8abeta)-
P037	60–57–1	2,7:3,6-Dimethanonaphth[2,3-b]oxirene, 3,4,5,6,9,9-hexachloro-1a,2,2a,3,6,6a,7,7a-octahydro-, (1aalpha,2beta,2aalpha,3beta,6beta,6aalpha,7beta, 7aalpha)-
P051	[1]72–20–8	2,7:3,6-Dimethanonaphth [2,3-b]oxirene, 3,4,5,6,9,9-hexachloro-1a,2,2a,3,6,6a,7,7a-octahydro-, (1aalpha,2beta,2abeta,3alpha,6alpha,6abeta,7beta, 7aalpha)-, & metabolites
P044	60–51–5	Dimethoate
P046	122–09–8	alpha,alpha-Dimethylphenethylamine
P191	644–64–4	Dimetilan.
P047	[1]534–52–1	4,6-Dinitro-o-cresol, & salts *
P048	51–28–5	2,4-Dinitrophenol
P020	88–85–7	Dinoseb
P085	152–16–9	Diphosphoramide, octamethyl-
P111	107–49–3	Diphosphoric acid, tetraethyl ester
P039	298–04–4	Disulfoton
P049	541–53–7	Dithiobiuret
P185	26419–73–8	1,3-Dithiolane-2-carboxaldehyde, 2,4-dimethyl-, O- [(methylamino)- carbonyl]oxime.
P050	115–29–7	Endosulfan
P088	145–73–3	Endothall
P051	72–20–8	Endrin
P051	72–20–8	Endrin, & metabolites
P042	51–43–4	Epinephrine
P031	460–19–5	Ethanedinitrile
P194	23135–22–0	Ethanimidothioc acid, 2-(dimethylamino)-N-[[(methylamino) carbonyl]oxy]-2-oxo-, methyl ester.
P066	16752–77–5	Ethanimidothioic acid, N-[[(methylamino)carbonyl]oxy]-, methyl ester
P101	107–12–0	Ethyl cyanide
P054	151–56–4	Ethyleneimine
P097	52–85–7	Famphur
P056	7782–41–4	Fluorine
P057	640–19–7	Fluoroacetamide
P058	62–74–8	Fluoroacetic acid, sodium salt
P198	23422–53–9	Formetanate hydrochloride.
P197	17702–57–7	Formparanate.
P065	628–86–4	Fulminic acid, mercury(2+) salt (R,T)
P059	76–44–8	Heptachlor
P062	757–58–4	Hexaethyl tetraphosphate
P116	79–19–6	Hydrazinecarbothioamide
P068	60–34–4	Hydrazine, methyl-
P063	74–90–8	Hydrocyanic acid
P063	74–90–8	Hydrogen cyanide
P096	7803–51–2	Hydrogen phosphide
P060	465–73–6	Isodrin
P192	119–38–0	Isolan.
P202	64–00–6	3-Isopropylphenyl N-methylcarbamate.
P007	2763–96–4	3(2H)-Isoxazolone, 5-(aminomethyl)-
P196	15339–36–3	Manganese, bis(dimethylcarbamodithioato-S,S')-,
P196	15339–36–3	Manganese dimethyldithiocarbamate.
P092	62–38–4	Mercury, (acetato-O)phenyl-
P065	628–86–4	Mercury fulminate (R,T)
P082	62–75–9	Methanamine, N-methyl-N-nitroso-
P064	624–83–9	Methane, isocyanato-
P016	542–88–1	Methane, oxybis[chloro-
P112	509–14–8	Methane, tetranitro- (R)
P118	75–70–7	Methanethiol, trichloro-
P198	23422–53–9	Methanimidamide, N,N-dimethyl-N'-[3-[[(methylamino)-carbonyl]oxy]phenyl]-, monohydrochloride.
P197	17702–57–7	Methanimidamide, N,N-dimethyl-N'-[2-methyl-4-[[(methylamino)carbonyl]oxy]phenyl]-
P050	115–29–7	6,9-Methano-2,4,3-benzodioxathiepin, 6,7,8,9,10,10-hexachloro-1,5,5a,6,9,9a-hexahydro-, 3-oxide
P059	76–44–8	4,7-Methano-1H-indene, 1,4,5,6,7,8,8-heptachloro-3a,4,7,7a-tetrahydro-
P199	2032–65–7	Methiocarb.
P066	16752–77–5	Methomyl
P068	60–34–4	Methyl hydrazine
P064	624–83–9	Methyl isocyanate
P069	75–86–5	2-Methyllactonitrile
P071	298–00–0	Methyl parathion
P190	1129–41–5	Metolcarb.

Hazardous waste No.	Chemical abstracts No.	Substance
P128	315-8-4	Mexacarbate.
P072	86-88-4	alpha-Naphthylthiourea
P073	13463-39-3	Nickel carbonyl
P073	13463-39-3	Nickel carbonyl $Ni(CO)_4$, (T-4)-
P074	557-19-7	Nickel cyanide
P074	557-19-7	Nickel cyanide $Ni(CN)_2$
P075	[1]54-11-5	Nicotine, & salts
P076	10102-43-9	Nitric oxide
P077	100-01-6	p-Nitroaniline
P078	10102-44-0	Nitrogen dioxide
P076	10102-43-9	Nitrogen oxide NO
P078	10102-44-0	Nitrogen oxide NO_2
P081	55-63-0	Nitroglycerine (R)
P082	62-75-9	N-Nitrosodimethylamine
P084	4549-40-0	N-Nitrosomethylvinylamine
P085	152-16-9	Octamethylpyrophosphoramide
P087	20816-12-0	Osmium oxide OsO_4, (T-4)-.
P087	20816-12-0	Osmium tetroxide
P088	145-73-3	7-Oxabicyclo[2.2.1]heptane-2,3-dicarboxylic acid
P194	23135-22-0	Oxamyl.
P089	56-38-2	Parathion
P034	131-89-5	Phenol, 2-cyclohexyl-4,6-dinitro-
P048	51-28-5	Phenol, 2,4-dinitro-
P047	[1]534-52-1	Phenol, 2-methyl-4,6-dinitro-, & salts
P020	88-85-7	Phenol, 2-(1-methylpropyl)-4,6-dinitro-
P009	131-74-8	Phenol, 2,4,6-trinitro-, ammonium salt (R)
P128	315-18-4	Phenol, 4-(dimethylamino)-3,5-dimethyl-, methylcarbamate (ester).
P199	2032-65-7	Phenol, (3,5-dimethyl-4-(methylthio)-, methylcarbamate
P202	64-00-6	Phenol, 3-(1-methylethyl)-, methyl carbamate.
P201	2631-37-0	Phenol, 3-methyl-5-(1-methylethyl)-, methyl carbamate.
P092	62-38-4	Phenylmercury acetate
P093	103-85-5	Phenylthiourea
P094	298-02-2	Phorate
P095	75-44-5	Phosgene
P096	7803-51-2	Phosphine
P041	311-45-5	Phosphoric acid, diethyl 4-nitrophenyl ester
P039	298-04-4	Phosphorodithioic acid, O,O-diethyl S-[2-(ethylthio)ethyl] ester
P094	298-02-2	Phosphorodithioic acid, O,O-diethyl S-[(ethylthio)methyl] ester
P044	60-51-5	Phosphorodithioic acid, O,O-dimethyl S-[2-(methylamino)-2-oxoethyl] ester
P043	55-91-4	Phosphorofluoridic acid, bis(1-methylethyl) ester
P089	56-38-2	Phosphorothioic acid, O,O-diethyl O-(4-nitrophenyl) ester
P040	297-97-2	Phosphorothioic acid, O,O-diethyl O-pyrazinyl ester
P097	52-85-7	Phosphorothioic acid, O-[4-[(dimethylamino)sulfonyl]phenyl] O,O-dimethyl ester
P071	298-00-0	Phosphorothioic acid, O,O,-dimethyl O-(4-nitrophenyl) ester
P204	57-47-6	Physostigmine.
P188	57-64-7	Physostigmine salicylate.
P110	78-00-2	Plumbane, tetraethyl-
P098	151-50-8	Potassium cyanide
P098	151-50-8	Potassium cyanide K(CN)
P099	506-61-6	Potassium silver cyanide
P201	2631-37-0	Promecarb
P070	116-06-3	Propanal, 2-methyl-2-(methylthio)-, O-[(methylamino)carbonyl]oxime
P203	1646-88-4	Propanal, 2-methyl-2-(methyl-sulfonyl)-, O-[(methylamino)carbonyl] oxime.
P101	107-12-0	Propanenitrile
P027	542-76-7	Propanenitrile, 3-chloro-
P069	75-86-5	Propanenitrile, 2-hydroxy-2-methyl-
P081	55-63-0	1,2,3-Propanetriol, trinitrate (R)
P017	598-31-2	2-Propanone, 1-bromo-
P102	107-19-7	Propargyl alcohol
P003	107-02-8	2-Propenal
P005	107-18-6	2-Propen-1-ol
P067	75-55-8	1,2-Propylenimine
P102	107-19-7	2-Propyn-1-ol
P008	504-24-5	4-Pyridinamine
P075	[1]54-11-5	Pyridine, 3-(1-methyl-2-pyrrolidinyl)-, (S)-, & salts
P204	57-47-6	Pyrrolo[2,3-b]indol-5-ol, 1,2,3,3a,8,8a-hexahydro-1,3a,8-trimethyl-, methylcarbamate (ester), (3aS-cis)-.
P114	12039-52-0	Selenious acid, dithallium(1+) salt

Hazardous waste No.	Chemical abstracts No.	Substance
P103	630-10-4	Selenourea
P104	506-64-9	Silver cyanide
P104	506-64-9	Silver cyanide Ag(CN)
P105	26628-22-8	Sodium azide
P106	143-33-9	Sodium cyanide
P106	143-33-9	Sodium cyanide Na(CN)
P108	[1]57-24-9	Strychnidin-10-one, & salts
P018	357-57-3	Strychnidin-10-one, 2,3-dimethoxy-
P108	[1]57-24-9	Strychnine, & salts
P115	7446-18-6	Sulfuric acid, dithallium(1+) salt
P109	3689-24-5	Tetraethyldithiopyrophosphate
P110	78-00-2	Tetraethyl lead
P111	107-49-3	Tetraethyl pyrophosphate
P112	509-14-8	Tetranitromethane (R)
P062	757-58-4	Tetraphosphoric acid, hexaethyl ester
P113	1314-32-5	Thallic oxide
P113	1314-32-5	Thallium oxide Tl_2O_3
P114	12039-52-0	Thallium(I) selenite
P115	7446-18-6	Thallium(I) sulfate
P109	3689-24-5	Thiodiphosphoric acid, tetraethyl ester
P045	39196-18-4	Thiofanox
P049	541-53-7	Thioimidodicarbonic diamide $[(H_2N)C(S)]_2NH$
P014	108-98-5	Thiophenol
P116	79-19-6	Thiosemicarbazide
P026	5344-82-1	Thiourea, (2-chlorophenyl)-
P072	86-88-4	Thiourea, 1-naphthalenyl-
P093	103-85-5	Thiourea, phenyl-
P185	26419-73-8	Tirpate.
P123	8001-35-2	Toxaphene
P118	75-70-7	Trichloromethanethiol
P119	7803-55-6	Vanadic acid, ammonium salt
P120	1314-62-1	Vanadium oxide V_2O_5
P120	1314-62-1	Vanadium pentoxide
P084	4549-40-0	Vinylamine, N-methyl-N-nitroso-
P001	[1]81-81-2	Warfarin, & salts, when present at concentrations greater than 0.3%
P205	137-30-4	Zinc, bis(dimethylcarbamodithioato-S,S')-,
P121	557-21-1	Zinc cyanide
P121	557-21-1	Zinc cyanide $Zn(CN)_2$
P122	1314-84-7	Zinc phosphide Zn_3P_2, when present at concentrations greater than 10% (R,T)
P205	137-30-4	Ziram.

[1] CAS Number given for parent compound only.

(f) The commercial chemical products, manfacturing chemical intermediates, or off-specification commercial chemical products referred to in paragraphs (a) through (d) of this section, are identified as toxic wastes (T), unless otherwise designated and are subject to the small quantity generator exclusion defined in § 261.5 (a) and (g).

[*Comment:* For the convenience of the regulated community, the primary hazardous properties of these materials have been indicated by the letters T (Toxicity), R (Reactivity), I (Ignitability) and C (Corrosivity). Absence of a letter indicates that the compound is only listed for toxicity.]

These wastes and their corresponding EPA Hazardous Waste Numbers are:

Hazardous waste No.	Chemical abstracts No.	Substance
U394	30558-43-1	A2213.
U001	75-07-0	Acetaldehyde (I)
U034	75-87-6	Acetaldehyde, trichloro-
U187	62-44-2	Acetamide, N-(4-ethoxyphenyl)-
U005	53-96-3	Acetamide, N-9H-fluoren-2-yl-
U240	[1]94-75-7	Acetic acid, (2,4-dichlorophenoxy)-, salts & esters
U112	141-78-6	Acetic acid ethyl ester (I)

Hazardous waste No.	Chemical abstracts No.	Substance
U144	301-04-2	Acetic acid, lead(2+) salt
U214	563-68-8	Acetic acid, thallium(1+) salt
see F027	93-76-5	Acetic acid, (2,4,5-trichlorophenoxy)-
U002	67-64-1	Acetone (I)
U003	75-05-8	Acetonitrile (I,T)
U004	98-86-2	Acetophenone
U005	53-96-3	2-Acetylaminofluorene
U006	75-36-5	Acetyl chloride (C,R,T)
U007	79-06-1	Acrylamide
U008	79-10-7	Acrylic acid (I)
U009	107-13-1	Acrylonitrile
U011	61-82-5	Amitrole
U012	62-53-3	Aniline (I,T)
U136	75-60-5	Arsinic acid, dimethyl-
U014	492-80-8	Auramine
U015	115-02-6	Azaserine
U010	50-07-7	Azirino(2',3':3,4)pyrrolo(1,2-a)indole-4,7-dione, 6-amino-8-[[(aminocarbonyl)oxy]methyl]-1,1a,2,8,8a,8b-hexahydro-8a-methoxy-5-methyl-, [1aS-(1aalpha, 8beta,8aalpha,8balpha)]-
U280	101-27-9	Barban.
U278	22781-23-3	Bendiocarb.
U364	22961-82-6	Bendiocarb phenol.
U271	17804-35-2	Benomyl.
U157	56-49-5	Benz[j]aceanthrylene, 1,2-dihydro-3-methyl-
U016	225-51-4	Benz[c]acridine
U017	98-87-3	Benzal chloride
U192	23950-58-5	Benzamide, 3,5-dichloro-N-(1,1-dimethyl-2-propynyl)-
U018	56-55-3	Benz[a]anthracene
U094	57-97-6	Benz[a]anthracene, 7,12-dimethyl-
U012	62-53-3	Benzenamine (I,T)
U014	492-80-8	Benzenamine, 4,4'-carbonimidoylbis[N,N-dimethyl-
U049	3165-93-3	Benzenamine, 4-chloro-2-methyl-, hydrochloride
U093	60-11-7	Benzenamine, N,N-dimethyl-4-(phenylazo)-
U328	95-53-4	Benzenamine, 2-methyl-
U353	106-49-0	Benzenamine, 4-methyl-
U158	101-14-4	Benzenamine, 4,4'-methylenebis[2-chloro-
U222	636-21-5	Benzenamine, 2-methyl-, hydrochloride
U181	99-55-8	Benzenamine, 2-methyl-5-nitro-
U019	71-43-2	Benzene (I,T)
U038	510-15-6	Benzeneacetic acid, 4-chloro-alpha-(4-chlorophenyl)-alpha-hydroxy-, ethyl ester
U030	101-55-3	Benzene, 1-bromo-4-phenoxy-
U035	305-03-3	Benzenebutanoic acid, 4-[bis(2-chloroethyl)amino]-
U037	108-90-7	Benzene, chloro-
U221	25376-45-8	Benzenediamine, ar-methyl-
U028	117-81-7	1,2-Benzenedicarboxylic acid, bis(2-ethylhexyl) ester
U069	84-74-2	1,2-Benzenedicarboxylic acid, dibutyl ester
U088	84-66-2	1,2-Benzenedicarboxylic acid, diethyl ester
U102	131-11-3	1,2-Benzenedicarboxylic acid, dimethyl ester
U107	117-84-0	1,2-Benzenedicarboxylic acid, dioctyl ester
U070	95-50-1	Benzene, 1,2-dichloro-
U071	541-73-1	Benzene, 1,3-dichloro-
U072	106-46-7	Benzene, 1,4-dichloro-
U060	72-54-8	Benzene, 1,1'-(2,2-dichloroethylidene)bis[4-chloro-
U017	98-87-3	Benzene, (dichloromethyl)-
U223	26471-62-5	Benzene, 1,3-diisocyanatomethyl- (R,T)
U239	1330-20-7	Benzene, dimethyl- (I,T)
U201	108-46-3	1,3-Benzenediol
U127	118-74-1	Benzene, hexachloro-
U056	110-82-7	Benzene, hexahydro- (I)
U220	108-88-3	Benzene, methyl-
U105	121-14-2	Benzene, 1-methyl-2,4-dinitro-
U106	606-20-2	Benzene, 2-methyl-1,3-dinitro-
U055	98-82-8	Benzene, (1-methylethyl)- (I)
U169	98-95-3	Benzene, nitro-
U183	608-93-5	Benzene, pentachloro-
U185	82-68-8	Benzene, pentachloronitro-
U020	98-09-9	Benzenesulfonic acid chloride (C,R)
U020	98-09-9	Benzenesulfonyl chloride (C,R)
U207	95-94-3	Benzene, 1,2,4,5-tetrachloro-
U061	50-29-3	Benzene, 1,1'-(2,2,2-trichloroethylidene)bis[4-chloro-
U247	72-43-5	Benzene, 1,1'-(2,2,2-trichloroethylidene)bis[4- methoxy-
U023	98-07-7	Benzene, (trichloromethyl)-

Hazardous waste No.	Chemical abstracts No.	Substance
U234	99-35-4	Benzene, 1,3,5-trinitro-
U021	92-87-5	Benzidine
U202	[1]81-07-2	1,2-Benzisothiazol-3(2H)-one, 1,1-dioxide, & salts
U278	22781-23-3	1,3-Benzodioxol-4-ol, 2,2-dimethyl-, methyl carbamate.
U364	22961-82-6	1,3-Benzodioxol-4-ol, 2,2-dimethyl-,
U203	94-59-7	1,3-Benzodioxole, 5-(2-propenyl)-
U141	120-58-1	1,3-Benzodioxole, 5-(1-propenyl)-
U367	1563-38-8	7-Benzofuranol, 2,3-dihydro-2,2-dimethyl-
U090	94-58-6	1,3-Benzodioxole, 5-propyl-
U064	189-55-9	Benzo[rst]pentaphene
U248	[1]81-81-2	2H-1-Benzopyran-2-one, 4-hydroxy-3-(3-oxo-1-phenyl-butyl)-, & salts, when present at concentrations of 0.3% or less
U022	50-32-8	Benzo[a]pyrene
U197	106-51-4	p-Benzoquinone
U023	98-07-7	Benzotrichloride (C,R,T)
U085	1464-53-5	2,2'-Bioxirane
U021	92-87-5	[1,1'-Biphenyl]-4,4'-diamine
U073	91-94-1	[1,1'-Biphenyl]-4,4'-diamine, 3,3'-dichloro-
U091	119-90-4	[1,1'-Biphenyl]-4,4'-diamine, 3,3'-dimethoxy-
U095	119-93-7	[1,1'-Biphenyl]-4,4'-diamine, 3,3'-dimethyl-
U225	75-25-2	Bromoform
U030	101-55-3	4-Bromophenyl phenyl ether
U128	87-68-3	1,3-Butadiene, 1,1,2,3,4,4-hexachloro-
U172	924-16-3	1-Butanamine, N-butyl-N-nitroso-
U031	71-36-3	1-Butanol (I)
U159	78-93-3	2-Butanone (I,T)
U160	1338-23-4	2-Butanone, peroxide (R,T)
U053	4170-30-3	2-Butenal
U074	764-41-0	2-Butene, 1,4-dichloro- (I,T)
U143	303-34-4	2-Butenoic acid, 2-methyl-, 7-[[2,3-dihydroxy-2-(1-methoxyethyl)-3-methyl-1-oxobutoxy]methyl]-2,3,5,7a-tetrahydro-1H-pyrrolizin-1-yl ester, [1S-[1alpha(Z),7(2S*,3R*),7aalpha]]-
U031	71-36-3	n-Butyl alcohol (I)
U136	75-60-5	Cacodylic acid
U032	13765-19-0	Calcium chromate
U372	10605-21-7	Carbamic acid, 1H-benzimidazol-2-yl, methyl ester.
U271	17804-35-2	Carbamic acid, [1-[(butylamino)carbonyl]-1H-benzimidazol-2-yl]-, methyl ester.
U280	101-27-9	Carbamic acid, (3-chlorophenyl)-, 4-chloro-2-butynyl ester.
U238	51-79-6	Carbamic acid, ethyl ester
U178	615-53-2	Carbamic acid, methylnitroso-, ethyl ester
U373	122-42-9	Carbamic acid, phenyl-, 1-methylethyl ester.
U409	23564-05-8	Carbamic acid, [1,2-phenylenebis (iminocarbonothioyl)]bis-, dimethyl ester.
U097	79-44-7	Carbamic chloride, dimethyl-
U389	2303-17-5	Carbamothioic acid, bis(1-methylethyl)-, S-(2,3,3-trichloro-2-propenyl) ester.
U387	52888-80-9	Carbamothioic acid, dipropyl-, S-(phenylmethyl) ester.
U114	[1]111-54-6	Carbamodithioic acid, 1,2-ethanediylbis-, salts & esters
U062	2303-16-4	Carbamothioic acid, bis(1-methylethyl)-, S-(2,3-dichloro-2-propenyl) ester
U279	63-25-2	Carbaryl.
U372	10605-21-7	Carbendazim.
U367	1563-38-8	Carbofuran phenol.
U215	6533-73-9	Carbonic acid, dithallium(1+) salt
U033	353-50-4	Carbonic difluoride
U156	79-22-1	Carbonochloridic acid, methyl ester (I,T)
U033	353-50-4	Carbon oxyfluoride (R,T)
U211	56-23-5	Carbon tetrachloride
U034	75-87-6	Chloral
U035	305-03-3	Chlorambucil
U036	57-74-9	Chlordane, alpha & gamma isomers
U026	494-03-1	Chlornaphazin
U037	108-90-7	Chlorobenzene
U038	510-15-6	Chlorobenzilate
U039	59-50-7	p-Chloro-m-cresol
U042	110-75-8	2-Chloroethyl vinyl ether
U044	67-66-3	Chloroform
U046	107-30-2	Chloromethyl methyl ether
U047	91-58-7	beta-Chloronaphthalene
U048	95-57-8	o-Chlorophenol
U049	3165-93-3	4-Chloro-o-toluidine, hydrochloride
U032	13765-19-0	Chromic acid H_2CrO_4, calcium salt
U050	218-01-9	Chrysene

Hazardous waste No.	Chemical abstracts No.	Substance
U051	.....	Creosote
U052	1319-77-3	Cresol (Cresylic acid)
U053	4170-30-3	Crotonaldehyde
U055	98-82-8	Cumene (I)
U246	506-68-3	Cyanogen bromide (CN)Br
U197	106-51-4	2,5-Cyclohexadiene-1,4-dione
U056	110-82-7	Cyclohexane (I)
U129	58-89-9	Cyclohexane, 1,2,3,4,5,6-hexachloro-, (1alpha,2alpha,3beta,4alpha,5alpha,6beta)-
U057	108-94-1	Cyclohexanone (I)
U130	77-47-4	1,3-Cyclopentadiene, 1,2,3,4,5,5-hexachloro-
U058	50-18-0	Cyclophosphamide
U240	[1]94-75-7	2,4-D, salts & esters
U059	20830-81-3	Daunomycin
U060	72-54-8	DDD
U061	50-29-3	DDT
U062	2303-16-4	Diallate
U063	53-70-3	Dibenz[a,h]anthracene
U064	189-55-9	Dibenzo[a,i]pyrene
U066	96-12-8	1,2-Dibromo-3-chloropropane
U069	84-74-2	Dibutyl phthalate
U070	95-50-1	o-Dichlorobenzene
U071	541-73-1	m-Dichlorobenzene
U072	106-46-7	p-Dichlorobenzene
U073	91-94-1	3,3'-Dichlorobenzidine
U074	764-41-0	1,4-Dichloro-2-butene (I,T)
U075	75-71-8	Dichlorodifluoromethane
U078	75-35-4	1,1-Dichloroethylene
U079	156-60-5	1,2-Dichloroethylene
U025	111-44-4	Dichloroethyl ether
U027	108-60-1	Dichloroisopropyl ether
U024	111-91-1	Dichloromethoxy ethane
U081	120-83-2	2,4-Dichlorophenol
U082	87-65-0	2,6-Dichlorophenol
U084	542-75-6	1,3-Dichloropropene
U085	1464-53-5	1,2:3,4-Diepoxybutane (I,T)
U108	123-91-1	1,4-Diethyleneoxide
U028	117-81-7	Diethylhexyl phthalate
U395	5952-26-1	Diethylene glycol, dicarbamate.
U086	1615-80-1	N,N'-Diethylhydrazine
U087	3288-58-2	O,O-Diethyl S-methyl dithiophosphate
U088	84-66-2	Diethyl phthalate
U089	56-53-1	Diethylstilbesterol
U090	94-58-6	Dihydrosafrole
U091	119-90-4	3,3'-Dimethoxybenzidine
U092	124-40-3	Dimethylamine (I)
U093	60-11-7	p-Dimethylaminoazobenzene
U094	57-97-6	7,12-Dimethylbenz[a]anthracene
U095	119-93-7	3,3'-Dimethylbenzidine
U096	80-15-9	alpha,alpha-Dimethylbenzylhydroperoxide (R)
U097	79-44-7	Dimethylcarbamoyl chloride
U098	57-14-7	1,1-Dimethylhydrazine
U099	540-73-8	1,2-Dimethylhydrazine
U101	105-67-9	2,4-Dimethylphenol
U102	131-11-3	Dimethyl phthalate
U103	77-78-1	Dimethyl sulfate
U105	121-14-2	2,4-Dinitrotoluene
U106	606-20-2	2,6-Dinitrotoluene
U107	117-84-0	Di-n-octyl phthalate
U108	123-91-1	1,4-Dioxane
U109	122-66-7	1,2-Diphenylhydrazine
U110	142-84-7	Dipropylamine (I)
U111	621-64-7	Di-n-propylnitrosamine
U041	106-89-8	Epichlorohydrin
U001	75-07-0	Ethanal (I)
U404	121-44-8	Ethanamine, N,N-diethyl-
U174	55-18-5	Ethanamine, N-ethyl-N-nitroso-
U155	91-80-5	1,2-Ethanediamine, N,N-dimethyl-N'-2-pyridinyl-N'-(2-thienylmethyl)-
U067	106-93-4	Ethane, 1,2-dibromo-
U076	75-34-3	Ethane, 1,1-dichloro-
U077	107-06-2	Ethane, 1,2-dichloro-
U131	67-72-1	Ethane, hexachloro-

Hazardous waste No.	Chemical abstracts No.	Substance
U024	111-91-1	Ethane, 1,1'-[methylenebis(oxy)]bis[2-chloro-
U117	60-29-7	Ethane, 1,1'-oxybis-(I)
U025	111-44-4	Ethane, 1,1'-oxybis[2-chloro-
U184	76-01-7	Ethane, pentachloro-
U208	630-20-6	Ethane, 1,1,1,2-tetrachloro-
U209	79-34-5	Ethane, 1,1,2,2-tetrachloro-
U218	62-55-5	Ethanethioamide
U226	71-55-6	Ethane, 1,1,1-trichloro-
U227	79-00-5	Ethane, 1,1,2-trichloro-
U410	59669-26-0	Ethanimidothioic acid, N,N'- [thiobis[(methylimino)carbonyloxy]]bis-, dimethyl ester
U394	30558-43-1	Ethanimidothioic acid, 2-(dimethylamino)-N-hydroxy-2-oxo-, methyl ester.
U359	110-80-5	Ethanol, 2-ethoxy-
U173	1116-54-7	Ethanol, 2,2'-(nitrosoimino)bis-
U395	5952-26-1	Ethanol, 2,2'-oxybis-, dicarbamate.
U004	98-86-2	Ethanone, 1-phenyl-
U043	75-01-4	Ethene, chloro-
U042	110-75-8	Ethene, (2-chloroethoxy)-
U078	75-35-4	Ethene, 1,1-dichloro-
U079	156-60-5	Ethene, 1,2-dichloro-, (E)-
U210	127-18-4	Ethene, tetrachloro-
U228	79-01-6	Ethene, trichloro-
U112	141-78-6	Ethyl acetate (I)
U113	140-88-5	Ethyl acrylate (I)
U238	51-79-6	Ethyl carbamate (urethane)
U117	60-29-7	Ethyl ether (I)
U114	111-54-6	Ethylenebisdithiocarbamic acid, salts & esters
U067	106-93-4	Ethylene dibromide
U077	107-06-2	Ethylene dichloride
U359	110-80-5	Ethylene glycol monoethyl ether
U115	75-21-8	Ethylene oxide (I,T)
U116	96-45-7	Ethylenethiourea
U076	75-34-3	Ethylidene dichloride
U118	97-63-2	Ethyl methacrylate
U119	62-50-0	Ethyl methanesulfonate
U120	206-44-0	Fluoranthene
U122	50-00-0	Formaldehyde
U123	64-18-6	Formic acid (C,T)
U124	110-00-9	Furan (I)
U125	98-01-1	2-Furancarboxaldehyde (I)
U147	108-31-6	2,5-Furandione
U213	109-99-9	Furan, tetrahydro-(I)
U125	98-01-1	Furfural (I)
U124	110-00-9	Furfuran (I)
U206	18883-66-4	Glucopyranose, 2-deoxy-2-(3-methyl-3-nitrosoureido)-, D-
U206	18883-66-4	D-Glucose, 2-deoxy-2-[[(methylnitrosoamino)-carbonyl]amino]-
U126	765-34-4	Glycidylaldehyde
U163	70-25-7	Guanidine, N-methyl-N'-nitro-N-nitroso-
U127	118-74-1	Hexachlorobenzene
U128	87-68-3	Hexachlorobutadiene
U130	77-47-4	Hexachlorocyclopentadiene
U131	67-72-1	Hexachloroethane
U132	70-30-4	Hexachlorophene
U243	1888-71-7	Hexachloropropene
U133	302-01-2	Hydrazine (R,T)
U086	1615-80-1	Hydrazine, 1,2-diethyl-
U098	57-14-7	Hydrazine, 1,1-dimethyl-
U099	540-73-8	Hydrazine, 1,2-dimethyl-
U109	122-66-7	Hydrazine, 1,2-diphenyl-
U134	7664-39-3	Hydrofluoric acid (C,T)
U134	7664-39-3	Hydrogen fluoride (C,T)
U135	7783-06-4	Hydrogen sulfide
U135	7783-06-4	Hydrogen sulfide H_2S
U096	80-15-9	Hydroperoxide, 1-methyl-1-phenylethyl- (R)
U116	96-45-7	2-Imidazolidinethione
U137	193-39-5	Indeno[1,2,3-cd]pyrene
U190	85-44-9	1,3-Isobenzofurandione
U140	78-83-1	Isobutyl alcohol (I,T)
U141	120-58-1	Isosafrole
U142	143-50-0	Kepone
U143	303-34-4	Lasiocarpine
U144	301-04-2	Lead acetate

Hazardous waste No.	Chemical abstracts No.	Substance
U146	1335-32-6	Lead, bis(acetato-O)tetrahydroxytri-
U145	7446-27-7	Lead phosphate
U146	1335-32-6	Lead subacetate
U129	58-89-9	Lindane
U163	70-25-7	MNNG
U147	108-31-6	Maleic anhydride
U148	123-33-1	Maleic hydrazide
U149	109-77-3	Malononitrile
U150	148-82-3	Melphalan
U151	7439-97-6	Mercury
U152	126-98-7	Methacrylonitrile (I, T)
U092	124-40-3	Methanamine, N-methyl- (I)
U029	74-83-9	Methane, bromo-
U045	74-87-3	Methane, chloro- (I, T)
U046	107-30-2	Methane, chloromethoxy-
U068	74-95-3	Methane, dibromo-
U080	75-09-2	Methane, dichloro-
U075	75-71-8	Methane, dichlorodifluoro-
U138	74-88-4	Methane, iodo-
U119	62-50-0	Methanesulfonic acid, ethyl ester
U211	56-23-5	Methane, tetrachloro-
U153	74-93-1	Methanethiol (I, T)
U225	75-25-2	Methane, tribromo-
U044	67-66-3	Methane, trichloro-
U121	75-69-4	Methane, trichlorofluoro-
U036	57-74-9	4,7-Methano-1H-indene, 1,2,4,5,6,7,8,8-octachloro-2,3,3a,4,7,7a-hexahydro-
U154	67-56-1	Methanol (I)
U155	91-80-5	Methapyrilene
U142	143-50-0	1,3,4-Metheno-2H-cyclobuta[cd]pentalen-2-one, 1,1a,3,3a,4,5,5,5a,5b,6-decachlorooctahydro-
U247	72-43-5	Methoxychlor
U154	67-56-1	Methyl alcohol (I)
U029	74-83-9	Methyl bromide
U186	504-60-9	1-Methylbutadiene (I)
U045	74-87-3	Methyl chloride (I,T)
U156	79-22-1	Methyl chlorocarbonate (I,T)
U226	71-55-6	Methyl chloroform
U157	56-49-5	3-Methylcholanthrene
U158	101-14-4	4,4'-Methylenebis(2-chloroaniline)
U068	74-95-3	Methylene bromide
U080	75-09-2	Methylene chloride
U159	78-93-3	Methyl ethyl ketone (MEK) (I,T)
U160	1338-23-4	Methyl ethyl ketone peroxide (R,T)
U138	74-88-4	Methyl iodide
U161	108-10-1	Methyl isobutyl ketone (I)
U162	80-62-6	Methyl methacrylate (I,T)
U161	108-10-1	4-Methyl-2-pentanone (I)
U164	56-04-2	Methylthiouracil
U010	50-07-7	Mitomycin C
U059	20830-81-3	5,12-Naphthacenedione, 8-acetyl-10-[(3-amino-2,3,6-trideoxy)-alpha-L-lyxo-hexopyranosyl)oxy]-7,8,9,10-tetrahydro-6,8,11-trihydroxy-1-methoxy-, (8S-cis)-
U167	134-32-7	1-Naphthalenamine
U168	91-59-8	2-Naphthalenamine
U026	494-03-1	Naphthalenamine, N,N'-bis(2-chloroethyl)-
U165	91-20-3	Naphthalene
U047	91-58-7	Naphthalene, 2-chloro-
U166	130-15-4	1,4-Naphthalenedione
U236	72-57-1	2,7-Naphthalenedisulfonic acid, 3,3'-[(3,3'-dimethyl[1,1'-biphenyl]-4,4'-diyl)bis(azo)bis[5-amino-4-hydroxy]-, tetrasodium salt
U279	63-25-2	1-Naphthalenol, methylcarbamate.
U166	130-15-4	1,4-Naphthoquinone
U167	134-32-7	alpha-Naphthylamine
U168	91-59-8	beta-Naphthylamine
U217	10102-45-1	Nitric acid, thallium(1+) salt
U169	98-95-3	Nitrobenzene (I,T)
U170	100-02-7	p-Nitrophenol
U171	79-46-9	2-Nitropropane (I,T)
U172	924-16-3	N-Nitrosodi-n-butylamine
U173	1116-54-7	N-Nitrosodiethanolamine
U174	55-18-5	N-Nitrosodiethylamine
U176	759-73-9	N-Nitroso-N-ethylurea
U177	684-93-5	N-Nitroso-N-methylurea
U178	615-53-2	N-Nitroso-N-methylurethane

Hazardous waste No.	Chemical abstracts No.	Substance
U179	100-75-4	N-Nitrosopiperidine
U180	930-55-2	N-Nitrosopyrrolidine
U181	99-55-8	5-Nitro-o-toluidine
U193	1120-71-4	1,2-Oxathiolane, 2,2-dioxide
U058	50-18-0	2H-1,3,2-Oxazaphosphorin-2-amine, N,N-bis(2-chloroethyl)tetrahydro-, 2-oxide
U115*	75-21-8	Oxirane (I,T)
U126	765-34-4	Oxiranecarboxyaldehyde
U041	106-89-8	Oxirane, (chloromethyl)-
2	123-63-7	Paraldehyde
U183	608-93-5	Pentachlorobenzene
U184	76-01-7	Pentachloroethane
U185	82-68-8	Pentachloronitrobenzene (PCNB)
See F027	87-86-5	Pentachlorophenol
U161	108-10-1	Pentanol, 4-methyl-
U186	504-60-9	1,3-Pentadiene (I)
U187	62-44-2	Phenacetin
U188	108-95-2	Phenol
U048	95-57-8	Phenol, 2-chloro-
U039	59-50-7	Phenol, 4-chloro-3-methyl-
U081	120-83-2	Phenol, 2,4-dichloro-
U082	87-65-0	Phenol, 2,6-dichloro-
U089	56-53-1	Phenol, 4,4'-(1,2-diethyl-1,2-ethenediyl)bis-, (E)-
U101	105-67-9	Phenol, 2,4-dimethyl-
U052	1319-77-3	Phenol, methyl-
U132	70-30-4	Phenol, 2,2'-methylenebis[3,4,6-trichloro-
U411	114-26-1	Phenol, 2-(1-methylethoxy)-, methylcarbamate.
U170	100-02-7	Phenol, 4-nitro-
See F027	87-86-5	Phenol, pentachloro-
See F027	58-90-2	Phenol, 2,3,4,6-tetrachloro-
See F027	95-95-4	Phenol, 2,4,5-trichloro-
See F027	88-06-2	Phenol, 2,4,6-trichloro-
U150	148-82-3	L-Phenylalanine, 4-[bis(2-chloroethyl)amino]-
U145	7446-27-7	Phosphoric acid, lead(2+) salt (2:3)
U087	3288-58-2	Phosphorodithioic acid, O,O-diethyl S-methyl ester
U189	1314-80-3	Phosphorus sulfide (R)
U190	85-44-9	Phthalic anhydride
U191	109-06-8	2-Picoline
U179	100-75-4	Piperidine, 1-nitroso-
U192	23950-58-5	Pronamide
U194	107-10-8	1-Propanamine (I,T)
U111	621-64-7	1-Propanamine, N-nitroso-N-propyl-
U110	142-84-7	1-Propanamine, N-propyl- (I)
U066	96-12-8	Propane, 1,2-dibromo-3-chloro-
U083	78-87-5	Propane, 1,2-dichloro-
U149	109-77-3	Propanedinitrile
U171	79-46-9	Propane, 2-nitro- (I,T)
U027	108-60-1	Propane, 2,2'-oxybis[2-chloro-
U193	1120-71-4	1,3-Propane sultone
See F027	93-72-1	Propanoic acid, 2-(2,4,5-trichlorophenoxy)-
U235	126-72-7	1-Propanol, 2,3-dibromo-, phosphate (3:1)
U140	78-83-1	1-Propanol, 2-methyl- (I,T)
U002	67-64-1	2-Propanone (I)
U007	79-06-1	2-Propenamide
U084	542-75-6	1-Propene, 1,3-dichloro-
U243	1888-71-7	1-Propene, 1,1,2,3,3,3-hexachloro-
U009	107-13-1	2-Propenenitrile
U152	126-98-7	2-Propenenitrile, 2-methyl- (I,T)
U008	79-10-7	2-Propenoic acid (I)
U113	140-88-5	2-Propenoic acid, ethyl ester (I)
U118	97-63-2	2-Propenoic acid, 2-methyl-, ethyl ester
U162	80-62-6	2-Propenoic acid, 2-methyl-, methyl ester (I,T)
U373	122-42-9	Propham.
U411	114-26-1	Propoxur.
U387	52888-80-9	Prosulfocarb
U194	107-10-8	n-Propylamine (I,T)

Hazardous waste No.	Chemical abstracts No.	Substance
U083	78-87-5	Propylene dichloride
U148	123-33-1	3,6-Pyridazinedione, 1,2-dihydro-
U196	110-86-1	Pyridine
U191	109-06-8	Pyridine, 2-methyl-
U237	66-75-1	2,4-(1H,3H)-Pyrimidinedione, 5-[bis(2-chloroethyl)amino]-
U164	56-04-2	4(1H)-Pyrimidinone, 2,3-dihydro-6-methyl-2-thioxo-
U180	930-55-2	Pyrrolidine, 1-nitroso-
U200	50-55-5	Reserpine
U201	108-46-3	Resorcinol
U202	[1]81-07-2	Saccharin, & salts
U203	94-59-7	Safrole
U204	7783-00-8	Selenious acid
U204	7783-00-8	Selenium dioxide
U205	7488-56-4	Selenium sulfide
U205	7488-56-4	Selenium sulfide SeS_2 (R,T)
U015	115-02-6	L-Serine, diazoacetate (ester)
See F027	93-72-1	Silvex (2,4,5-TP)
U206	18883-66-4	Streptozotocin
U103	77-78-1	Sulfuric acid, dimethyl ester
U189	1314-80-3	Sulfur phosphide (R)
See F027	93-76-5	2,4,5-T
U207	95-94-3	1,2,4,5-Tetrachlorobenzene
U208	630-20-6	1,1,1,2-Tetrachloroethane
U209	79-34-5	1,1,2,2-Tetrachloroethane
U210	127-18-4	Tetrachloroethylene
See F027	58-90-2	2,3,4,6-Tetrachlorophenol
U213	109-99-9	Tetrahydrofuran (I)
U214	563-68-8	Thallium(I) acetate
U215	6533-73-9	Thallium(I) carbonate
U216	7791-12-0	Thallium(I) chloride
U216	7791-12-0	Thallium chloride TlCl
U217	10102-45-1	Thallium(I) nitrate
U218	62-55-5	Thioacetamide
U410	59669-26-0	Thiodicarb.
U153	74-93-1	Thiomethanol (I,T)
U244	137-26-8	Thioperoxydicarbonic diamide $[(H_2N)C(S)]_2S_2$, tetramethyl-
U409	23564-05-8	Thiophanate-methyl.
U219	62-56-6	Thiourea
U244	137-26-8	Thiram
U220	108-88-3	Toluene
U221	25376-45-8	Toluenediamine
U223	26471-62-5	Toluene diisocyanate (R,T)
U328	95-53-4	o-Toluidine
U353	106-49-0	p-Toluidine
U222	636-21-5	o-Toluidine hydrochloride
U389	2303-17-5	Triallate.
U011	61-82-5	1H-1,2,4-Triazol-3-amine
U227	79-00-5	1,1,2-Trichloroethane
U228	79-01-6	Trichloroethylene
U121	75-69-4	Trichloromonofluoromethane
See F027	95-95-4	2,4,5-Trichlorophenol
See F027	88-06-2	2,4,6-Trichlorophenol
U404	121-44-8	Triethylamine.
U234	99-35-4	1,3,5-Trinitrobenzene (R,T)
U182	123-63-7	1,3,5-Trioxane, 2,4,6-trimethyl-
U235	126-72-7	Tris(2,3-dibromopropyl) phosphate
U236	72-57-1	Trypan blue
U237	66-75-1	Uracil mustard
U176	759-73-9	Urea, N-ethyl-N-nitroso-
U177	684-93-5	Urea, N-methyl-N-nitroso-
U043	75-01-4	Vinyl chloride
U248	[1]81-81-2	Warfarin, & salts, when present at concentrations of 0.3% or less
U239	1330-20-7	Xylene (I)
U200	50-55-5	Yohimban-16-carboxylic acid, 11,17-dimethoxy-18-[(3,4,5-trimethoxybenzoyl)oxy]-, methyl ester, (3beta,16beta,17alpha,18beta,20alpha)-

APPENDIX 2

HAZARDOUS WASTE PROFILE SHEET

HAZARDOUS WASTE PROFILE SHEET

ECTION 1 To be completed by Generator | NSWC WASTE STREAM NO.:

1. BUILDING # GENERATING WASTE	2. POC	4. PHONE
	3. TITLE	

5. Describe the industrial operations that generated your hazardous waste. Include information on the steps taken in the operation, the machinery, and materials used, and the inputs and outputs of the operation. Attach MSDSs for all chemicals used. If more than one chemical is mixed or if other materials are contaminated in the process, they may need to be profiled separately (e.g., liquids kept separate from solids).

6. MATERIAL COMPOSITION:

COMPONENT	%

% Must Total 100

7. Mark any of the following toxic contaminants if there is any chance that they may be found in the waste. Provide concentrations, if known, in mg/L

CONTAMINANT	EPA #	(mg/L)		CONTAMINANT	EPA #	(mg/L)
ARSENIC	D004			HEXACHLOROBENZENE	D032	
BARIUM	D005			HEXACHLORO-1,3-BUTADIENE	D033	
BENZENE	D018			HEXACHLOROETHANE	D034	
CADMIUM	D006			LEAD	D008	
CARBON TETRACHLORIDE	D019			LINDANE	D013	
CHLORDANE	D020			MERCURY	D009	
CHLOROBENZENE	D021			METHOXYCHLOR	D014	
CHLOROFORM	D022			METHYL ETHYL KETONE	D035	
CHROMIUM	D007			NITROBENZENE	D036	
O-CRESOL	D023			PENTACHLOROPHENOL	D037	
M-CRESOL	D024			PYRIDINE	D038	
P-CRESOL	D025			SELENIUM	D010	
CRESOL	D026			SILVER	D011	
2,4-D	D016			TETRACHLOROETHYLENE	D039	
1,4-DICHLOROBENZENE	D027			TOXOPHENE	D015	
1,2-DICHLOROETHANE	D028			TRICHLOROETHYLENE	D040	
1,1-DICHLOROETHYLENE	D029			2,4,5-TRICHLOROPHENOL	D041	
2,4-DINITROTOLUENE	D030			2,4,6-TRICHLOROPHENOL	D042	
ENDRIN	D012			2,45-TP (SILVEX)	D017	
HEPTACHLOR (AND ITS HYDROXIDE)	D031			VINYL CHLORIDE	D043	

UNITS (GAL, LBS, DRUMS)	(Week, Month, or Year)
8. PROJECTED VOLUME:	FREQUENCY:

9. I, _____ (Print or Type Name), Hereby certify that all information submitted in this and all attached documents is, to the best of my knowledge, an accurate representation of the waste turned in to Code 095, and all known or suspected hazards have been disclosed.

SIGNATURE OF GENERATOR'S REPRESENTATIVE	DATE

HAZARDOUS WASTE PROFILE SHEET cont'd

SECTION 2 To be completed by Code 095	NSWC WASTE STREAM NO.:
1. Sample Analysis: Yes ___ No ___	Date Sampled: ___/___/___

2. RCRA CHARACTERISTICS

PHYSICAL STATE: ___ SOLID ___ LIQUID ___ SEMI-SOLID ___ GAS ___ OTHER

TREATMENT GROUP: ___ WASTEWATER ___ NON-WASTEWATER

___ IGNITABLE (D001)
FLASH POINT (F) _____
___ HIGH TOC (>10%)
___ LOW TOC (<10%)
___ CORROSIVE (D002)
pH _____
___ CORRODES STEEL

___ REACTIVE (D003)
___ WATER REACTIVE
___ CYANIDE REACTIVE
___ SULFIDE REACTIVE
___ TOXICITY CHARACTERISTIC

3. SHIPPING INFORMATION

DOT HAZARDOUS MATERIAL: ___ Yes ___ No

PROPER SHIPPING NAME:

HAZARD CLASS: | U.N. or N.A. NO.:

METHOD OF SHIPMENT: ___ BULK ___ DRUM ___ OTHER:

ADDITIONAL DESCRIPTION:

SECTION 3 To be completed by Code 095

____ The material evaluated by Code 095 has been determined to be a SOLID WASTE and does not require management as hazardous waste. This material may require special handling and additional management instruction will be provided by Code 095.

____ This material has been characterized by Code 095 as a HAZARDOUS WASTE. All specific requirements of RCRA and NSWCINST 5090.2A apply.

SPECIFIC LABEL REQUIREMENTS ARE:

PROFILE #: _____
CONTENTS: _____
CODE: _____

SIGNATURE OF CODE 095'S REPRESENTATIVE	DATE

NAVSURFWARCENDIV CRANE 6280/11 (REV 6/99) (BACK)

APPENDIX 3

WASTE ACCUMULATION SITE WEEKLY INSPECTION CHECKLIST

WASTE ACCUMULATION SITE WEEKLY INSPECTION CHECKLIST

BUILDING	DATE OF INSPECTION	INSPECTOR'S NAME	INSPECTOR'S SIGNATURE

ITEMS	STATUS Acceptable (A) Unacceptable (U)	COMMENTS & CORRECTIVE MEASURES
• Containers are in good condition (not bulging, corroded, leaking, dented, etc.)		
• Wastes are placed in an approved container, compatible with the waste		
• Container is properly labelled and filled out properly		
• Containers are closed except when adding waste		
• Containers of incompatible wastes are separated by berm, dike, wall, or other device		
• Containers are stored at the designated accumulation site		
• Absorbent materials are available to contain leaks or spills		
• (Type I area only) Less than 55gal. of waste on–site, or if greater than 55gal., then on hand for less than 3 days		
• (Type II area only) No waste stored for greater than 90 days from the start of generation		
• (Type II area only) Aisle space of 30 to 39 inches maintained around containers of waste		

NAVSURFWARCENDIV CRANE 6280/1 (REV. 6-25)

APPENDIX 4

LABORATORY WASTE INVENTORY FORM

LABORATORY WASTE INVENTORY

CHEMICAL NAME	NO. OF CONT.	APPROXIMATE QUANTITY (lb, gal, oz, etc.)	TYPE OF CONTAINER (Glass, Plastic, Metal, Cardboard)	BLDG.	095 COMMENTS

Signature *(Generator)*	BUILDING	PHONE EXT.	CODE	DATE

JOB COMPLETED BY

SIGNATURE *(095 Approval)*

DISTRIBUTION

Send White Copy to Bldg. 3140, Code 095, to arrange for collection of waste. Yellow Copy should accompany material

NAVSUPPWARCENDIV CRANE 5280/5 (REV. 6-80)

APPENDIX 5

CHECKLIST FOR EXPLOSIVE HAZARDOUS WASTE MANAGEMENT, TYPE 1-X ACCUMULATION AREA

CHECKLIST FOR EXPLOSIVE HAZARDOUS WASTE MANAGEMENT,
TYPE 1-X ACCUMULATION AREA
LESS THAN 55 GALLONS AT OR NEAR THE POINT OF GENERATION
CONDITIONS FOR EXPLOSIVE HAZARDOUS WASTE MANAGEMENT

TO BE IN COMPLIANCE, THE FOLLOWING STANDARDS MUST BE MET:

1. Explosive Hazardous Waste (EHW) must be placed into a container in good condition.

2. The container must be made of materials compatible to the EHW being collected.

3. The container must be placed at or near the point of generation and in an area approved by the EPD. The storage areas holding EHW must also be approved by the Explosives Safety Department.

4. At a minimum of once per day, move all EHW or pyrotechnic scrap from the process or generating location to the designated storage location to await movement to the treatment site. An exception to this requirement could be granted, but it must be documented in the applicable SOP. In no case shall quantities greater than 55 gallons be held for more than three days without being taken to the treatment site.

5. Properly close the container used to collect EHW and affix the "Explosive Hazardous Waste Scrap" label. The label must be affixed to the container as soon as the first increment of waste is placed into the container. **NOTE: Do not tighten the container such that its contents would cause a rupture or release.**

6. Secondary containment must be provided for any liquid EHW containers under the operator's control.

7. The storage/collection area must also provide protection from precipitation run-on and/or run-off.

8. The storage/collection area must be under the operator's control.

9. Proper signs must be posted at the storage/collection area. Required signs should include the words "No

Smoking" and "Hazardous Waste" to be displayed at the entrance to the storage/collection area. In addition, an Explosive Hazardous Waste Site Rules sign shall also be posted at this area. An example of this sign can be found in Appendix 7 of the Hazardous Waste Management Plan, NAVSURFWARCENDIVINST 5090.2B.

10. The operator must maintain records that track the materials into the storage location and to the next management facility (which in most cases will be the Ammunition Burning Ground or the Demolition Range).

11. Additional record keeping required includes a site diagram that displays the storage location, location of emergency response equipment and the location of any communication devices that may be used to summon assistance in case of emergency. A list of telephone numbers and/or radio call signs must be provided and maintained with the communication devices.

OTHER INFORMATION:

A. Assistance with obtaining signs and labels will be provided by the EPD, upon notification.

B. This Checklist is part of the Hazardous Waste Management Plan, NAVSURFWARCENDIVINST 5090.2B. Additional information regarding EHW management can be found in that plan.

C. For the convenience of the user of this checklist, the following definitions are provided to delineate between Explosive Hazardous Waste and Explosive Contaminated Materials:

1. EXPLOSIVE HAZARDOUS WASTE (EHW): Waste generated from an explosive operation that meets the U.S. Environmental Protection Agency's (USEPA) definition of a reactive waste, such as bulk explosive scrap, pyrotechnics, and materials with a sufficient amount of explosive residue to pose a safety hazard. Non-functioned munitions items that have failed quality or safety inspections and are determined to be an explosive safety hazard are also considered EHW.

2. <u>Explosive Contaminated Materials (ECM)</u>: Materials that have been in contact with explosives such as cardboard, floor sweepings, rags, wood, spent time fuses, functioned items, and metal will not be considered EHW unless a significant amount of raw explosives are present and pose an explosive hazard. Since ECMs do not meet the USEPA's definition of a reactive waste, they will not be considered as EHW and will be flashed at the ABG for safety reasons only. OP 5 requires that all ECM is thermally treated to assure there is no possibility of an explosive hazard present. These items will still be handled according to current procedures with the exception of labeling and record keeping requirements. The ECM will be labeled as "OP 5 ITEMS AWAITING SAFETY FLASHING" and will not be under the standard requirements of EHW.

APPENDIX 6

EXPLOSIVE HAZARDOUS WASTE SCRAP LABEL

EXPLOSIVE

HAZARDOUS WASTE SCRAP

CONTENTS:

CONTACT:

DATE:

APPENDIX 7

EXPLOSIVE HAZARDOUS WASTE SITE RULES SIGN

EXPLOSIVE HAZARDOUS WASTE SITE RULES

- AT THIS SITE, STORE ONLY EXPLOSIVE HAZARDOUS WASTE
- ANY WASTE PLACED AT THIS SITE MUST BE REMOVED WITHIN THE TIME SPECIFIED IN THE PROCESS SOP
- RECORD IN THE SITE LOG ALL WASTES PLACED INTO AND REMOVED FROM THIS SITE

NO SMOKING

APPENDIX 8

CONTAMINATED SCRAP MANIFEST FORM

CONTAMINATED SCRAP MANIFEST

The following material is hereby certified as being contaminated with explosives and/or pyrotechnics

TYPE OF SCRAP	TYPE OF CONTAINER	TYPE OF CONTAMINATION	BUILDING/AREA AND CODE	APPROXIMATE QUANTITY (lb.)

*Indicate if material is mixed with water or other chemical solvents. Identify the specific chemical solvent used.

SIGNATURE *(Generator or Facility Supervisor-Extension No.)*	CODE	DATE

SIGNATURE *(Transporter)*	ORGANIZATION	DATE

SIGNATURE *(Operator at Treatment Facility)*	TREATMENT FACILITY	DATE

NAVSURFWARCENDIV CRANE 6280/4 (REV. 6-99)
DISTRIBUTION
The following copies shall be withdrawn by the respective personnel and sent to Code 095, Building 3260, within 5 work days.

Gold - Generator; Yellow - Transporter; White - Treatment Facility Operator

APPENDIX 9

SPENT MUNITIONS ITEMS SIGN

SPENT MUNITIONS ITEMS

- Per NAVSEA OP 5 Para. 13 - 18.1
- Items that require flash burning shall be marked, "OP 5 items awaiting safety flashing"
- *NOTE: Items that do not require additional flashing should be marked "INERT" and transferred for scrap metal recovery or disposed of as solid waste*

APPENDIX 10

OP 5 ITEMS WAITING SAFETY FLASHING LABEL

OP 5 ITEMS

AWAITING SAFETY FLASHING

CONTENTS:

CONTACT:

DATE:

APPENDIX 11

DEFENSE LOGISTICS AGENCY HAZARDOUS WASTE PROFILE SHEET

2 3 JUL 1994

HAZARDOUS WASTE PROFILE SHEET

PART I

GENERAL INFORMATION

WASTE PROFILE NO. _____

1. GENERATOR NAME

2. FACILITY ADDRESS

3. GENERATOR USEPA ID

4. GENERATOR STATE ID

5. ZIP CODE

6. TECHNICAL CONTACT

7. TITLE

PHONE ( )

8. 1. NAME OF WASTE _____

2. USEPA/or/STATE WASTE CODE(S) _____

3. PROCESS GENERATING WASTE _____

4. PROJECTED ANNUAL VOLUME/UNITS _____ / _____ 5. MODE OF COLLECTION _____

6. IS THIS WASTE A DIOXIN LISTED WASTE AS DEFINED IN 40 CFR 261.31 (e.g., F020, F021, F022, F023, F026, F027, OR F028)? ☐ YES ☐ NO

7. IS THIS WASTE RESTRICTED FROM LAND DISPOSAL (40 CFR 268)? ☐ YES ☐ NO
HAS AN EXEMPTION BEEN GRANTED? ☐ YES ☐ NO
DOES THE WASTE MEET APPLICABLE TREATMENT STANDARDS? ☐ YES ☐ NO REFERENCE STANDARDS _____

PART II

1. MATERIAL CHARACTERIZATION
(OPTIONAL-NOT REQUIRED DATA)

COLOR _____

DENSITY _____ BTU/LB _____

TOTAL SOLIDS _____ ASH CONTENT _____

LAYERING: ☐ MULTILAYERED ☐ BILAYERED ☐ SINGLE PHASE

2. RCRA CHARACTERISTICS

PHYSICAL STATE: ☐ SOLID ☐ LIQUID ☐ SEMI-SOLID ☐ GAS ☐ OTHER

TREATMENT GROUP: ☐ WASTEWATER ☐ NON-WASTEWATER

☐ IGNITABLE (D001)
FLASH POINT (F) _____
☐ HIGH TOC (> 10%)
☐ LOW TOC (< 10%)

☐ REACTIVE (D003)
☐ WATER REACTIVE
☐ CYANIDE REACTIVE
☐ SULFIDE REACTIVE

☐ CORROSIVE (D002)
pH _____
☐ CORRODES STEEL

☐ TOXICITY CHARACTERISTIC
(SEE REVERSE FOR LISTING)

3. CHEMICAL COMPOSITION (ppm or mg/L)

COPPER _____ PHENOLICS _____
NICKEL _____ TOTAL HALOGENS _____
ZINC _____ VOLATILE ORGANICS _____
CHROMIUM-HEX _____ PCBs _____
(OTHER) _____

NOTE: EXPLOSIVES, SHOCK SENSITIVE, PYROPHORIC, RADIOACTIVE, AND ETIOLOGICAL WASTE NORMALLY ARE NOT ACCEPTED BY THE DRMO.

4. MATERIAL COMPOSITION

COMPONENT	CONCENTRATION	RANGE

TOTAL _____ 100%

5. SHIPPING INFORMATION

DOT HAZARDOUS MATERIAL? ☐ YES ☐ NO

PROPER SHIPPING NAME _____

HAZARD CLASS _____ U.N. or N.A. NO. _____
ADDITIONAL DESCRIPTION _____
METHOD OF SHIPMENT ☐ BULK ☐ DRUM ☐ OTHER: _____
CERCLA REPORTABLE QUANTITY (RQ) _____
EMERGENCY RESPONSE GUIDE PAGE
DOT PUBLICATION 5800.4 PAGE NO. _____ EDITION (YR) _____
SPECIAL HANDLING INFORMATION _____

6. GENERATOR CERTIFICATION

BASIS FOR INFORMATION
☐ CHEMICAL ANALYSIS *(ATTACH TEST RESULTS)*
☐ USER KNOWLEDGE *(ATTACH SUPPORTING DOCUMENTS - Explain how and why these documents comply with RCRA requirements)* _____

_____ *(Print or Type Name)*, HEREBY CERTIFY THAT ALL INFORMATION SUBMITTED IN THIS AND ALL ATTACHED DOCUMENTS IS TO THE BEST OF MY KNOWLEDGE AN ACCURATE REPRESENTATION OF THE WASTE TURNED IN TO THE DRMO. ALL KNOWN OR SUSPECTED HAZARDS HAVE BEEN DISCLOSED.

SIGNATURE OF GENERATOR'S REPRESENTATIVE

DATE

TOXICITY CHARACTERISTIC LIST

EFFECTIVE 26 SEP 90 - LARGE QUANTITY GENERATORS
29 MAR 91 - SMALL QUANTITY GENERATORS

CONTAMINANT	EPA HW No.	(mg/L)	CONTAMINANT	EPA HW No.	(mg/L)
☐ ARSENIC	D004	_____	☐ HEXACHLORO-1,3,-BUTADIENE	D033	_____
☐ BARIUM	D005	_____	☐ HEXACHLOROETHANE	D034	_____
☐ BENZENE	D018	_____	☐ LEAD	D008	_____
☐ CADMIUM	D006	_____	☐ LINDANE	D013	_____
☐ CARBON TETRACHLORIDE	D019	_____	☐ MERCURY	D009	_____
☐ CHLORDANE	D020	_____	☐ METHOXYCHLOR	D014	_____
☐ CHLOROBENZENE	D021	_____	☐ METHYL ETHYL KETONE	D035	_____
☐ CHLOROFORM	D022	_____	☐ NITROBENZENE	D036	_____
☐ CHROMIUM	D007	_____	☐ PENTACHLOROPHENOL	D037	_____
☐ O-CRESOL	D023	_____	☐ PYRIDINE	D038	_____
☐ M-CRESOL	D024	_____	☐ SELENIUM	D010	_____
☐ P-CRESOL	D026	_____	☐ SILVER	D011	_____
☐ CRESOL	D026	_____	☐ TETRACHLOROETHYLENE	D039	_____
☐ 2,4-D	D016	_____	☐ TOXOPHENE	D015	_____
☐ 1,4-DICHLOROBENZENE	D027	_____	☐ TRICHLOROETHYLENE	D040	_____
☐ 1,2-DICHLOROETHANE	D028	_____	☐ 2,4,5-TRICHLOROPHENOL	D041	_____
☐ 1,1-DICHLOROETHYLENE	D029	_____	☐ 2,4,6-TRICHLOROPHENOL	D042	_____
☐ 2,4-DINITROTOLUENE	D030	_____	☐ 2,4S-TP (SILVEX)	D017	_____
☐ ENDRIN	D012	_____	☐ VINYL CHLORIDE	D043	_____
☐ HEPTACHLOR (AND ITS HYDROXIDE)	D031	_____			
☐ HEXACHLOROBENZENE	D032	_____			

PART III

FOR DRMO USE ONLY

DRMO VERIFICATION

1. DATE VERIFIED _____

2. RESULTS ☐ *ATTACHED*

pH _____ FLASH POINT _____ SPECIFIC GRAVITY _____ HALIDES (TOX) _____

REACTIVITY: WATER REACTIVITY _____ CYANIDES _____ SULFIDES _____

TCLP _____

APPENDIX 12

HAZARDOUS WASTE LABEL

HAZARDOUS WASTE

CRANE DIVISION
NAVAL SURFACE WARFARE CENTER

WASTE DESCRIPTION

SERIAL NUMBER

NSWC WASTE PROFILE NUMBER

CONTACT: EXT

START DATE FILL DATE

APPENDIX 13

HAZARDOUS WASTE GENERATION LOG

HAZARDOUS WASTE GENERATION LOG

SITE NUMBER_____

CONTAINER CONTENTS	LABEL SERIAL NUMBER	DATE FILL STARTED	WASTE PROFILE NUMBER	DATE FILL FINAL	TRANSFER SERIAL NUMBER	DATE TRANSFER REQUESTED	DATE TRANSFER ACCOMPLISHED

NAVSURFWARCENDIV CRANE 6280/2 (REV. 6-25)

APPENDIX 14

NON-REGULATED WASTE LABEL

NON-REGULATED WASTE

CRANE DIVISION
NAVAL SURFACE WARFARE CENTER

WASTE DESCRIPTION

SERIAL NUMBER

NSWC WASTE PROFILE NUMBER

CONTACT EXT

START DATE FILL DATE

APPENDIX 15

HAZARDOUS WASTE PROGRAM CONTROL RECORD

HAZARDOUS WASTE PROGRAM CONTROL RECORD

SERIAL NO.

EMPTY DRUMS DELIVERY *(Number and Type)*

CONTAINERS PICKUP *(Number and Size)*

WASTE TYPE

LOCATION

CONTACT *(Name and Telephone Extension)*

ADDITIONAL INFORMATION

MESSAGE RECEIVED BY

DATE

JOB COMPLETED BY

DATE

NAVSURFWARCENDIV CRANE 6260/6 (REV. 6-99)

HAZARDOUS WASTE PROGRAM CONTROL RECORD

SERIAL NO.

EMPTY DRUMS DELIVERY *(Number and Type)*

CONTAINERS PICKUP *(Number and Size)*

WASTE TYPE

LOCATION

CONTACT *(Name and Telephone Extension)*

ADDITIONAL INFORMATION

MESSAGE RECEIVED BY

DATE

JOB COMPLETED BY

DATE

APPENDIX 16

CENTRAL STORAGE FACILITY LOG SHEET

CENTRAL STORAGE FACILITY LOG SHEET
BUILDING 2993

DRM NUMBER	CONTAINER SIZE	POUNDS	PHYSICAL FORM	GENERATOR	COMPOSITION	EPA HW-ID	NSWC VSNO	DATE IN STORAGE	B-2993 LOCATION	LAND BAND NOTIFICATION	CHARGE NUMBER	
			LIQUID							YES		
			SOLID							NO		
			LIQUID							YES		
			SOLID							NO		
			LIQUID							YES		
			SOLID							NO		
			LIQUID							YES		
			SOLID							NO		
			LIQUID							YES		
			SOLID							NO		
			LIQUID							YES		
			SOLID							NO		
			LIQUID							YES		
			SOLID							NO		
			LIQUID							YES		
			SOLID							NO		
			LIQUID							YES		
			SOLID							NO		
			LIQUID							YES		
			SOLID							NO		
			LIQUID							YES		
			SOLID							NO		
			LIQUID							YES		
			SOLID							NO		

NAVSURFWARCENDIV CRANE 6290/6 (REV. 6-25)

APPENDIX 17

CENTRAL STORAGE FACILITY WEEKLY INSPECTION LOG

ENVIRONMENTAL PROTECTION WEEKLY INSPECTION LOG

CENTRAL STORAGE FACILITY

ITEM	REASON FOR INSPECTION	STATUS. ACCEPTABLE (A) UNACCEPTABLE (U)	OBSERVATIONS	DATE AND NATURE OF REPAIRS AND/OR REMEDIAL ACTION
CONTAINERS	LEAKING, DAMAGED RUST CORROSION			
LABELS	MISSING, IMPROPERLY COMPLETED			
AISLE SPACE	PROPERLY SPACED			
WARNING SIGNS	MISSING, DAMAGED			
FLOOR/SUMPS	FREE FROM LIQUIDS, CRACKS, DAMAGED			
CURB	DAMAGED			
BUILDING	WALLS, ROOF DAMAGED			
HOUSEKEEPING	AREA LITTERED			
DRUM DOLLY	BROKEN			
ABSORBENT	OUT OF STOCK			
TRANSFER PUMPS/HOSE	LEAKS, OPERABILITY			
EYE WASH/SHOWERS	FUNCTION			

ENV NMENTAL PROTECTION WEEKLY INSPECTION LOG
CENTRAL STORAGE FACILITY

INSPECTOR'S NAME (Printed/Title)	SIGNATURE (Inspector's)
DATE OF INSPECTION (Month, Day, Year)	TIME OF INSPECTION (Military Time)

ITEM	REASON FOR INSPECTION	STATUS ACCEPTABLE (A) UNACCEPTABLE (U)	OBSERVATIONS	DATE AND NATURE OF REPAIRS AND/OR REMEDIAL ACTION
RADIO ALARM SYSTEM TELEPHONE	OPERABILITY			
FIRE EXTINGUISHERS	NEED RECHARGING			
GATE LOCK	DEFECTIVE			
GATES	DAMAGED; DOES NOT CLOSE PROPERLY			
DOOR LOCKS	DEFECTIVE			
DOORS	DAMAGED			
COMPATIBLE STORAGE	NONCOMPATIBLE STORED TOGETHER			

NAVSURFWARCENDIVCRANE INSTRUCTION 4855.20

From: Commander

Subj: CALIBRATION PROGRAM

Ref: (a) NAVSEAINST 4734.1A, NAVSEA Metrology and Calibration Program

Encl: (1) Calibration Program

1. Purpose. To delineate policy, responsibilities and procedures for the Calibration Program at Crane Division, Naval Surface Warfare Center (NAVSURFWARCENDIV Crane).

2. Cancellation. NAVWPNSUPPCENINST 4355.17K.

3. Scope. This instruction applies to NAVSURFWARCENDIV Crane and tenant activities at the Crane site.

4. Policy

a. Test and measurement equipment (TME) used to make, record or generate quantitative measurements or data in the development, production, inspection or testing of material shall be in a valid and current state of calibration.

b. The Calibration Laboratory and Repair Branch (Code 6034) is the only Division activity authorized to affix official Navy Calibration servicing labels and tags to TME. Only those calibration labels or tags approved by the Calibration Laboratory will be authorized for use on TME.

5. Action

a. Calibration Laboratory and Repair Branch (Code 6034) shall manage, operate and maintain a certified Calibration Laboratory in accordance with reference (a) and the applicable Calibration Program requirements identified in enclosure (1).

b. Directorate Directors and Heads of Tenant Activities shall ensure that the applicable requirements of enclosure (1) are implemented.

c. The Quality Management Office (Code QMO) shall maintain the Calibration Program instruction and annually review its implementation.

6. Forms. The following forms are required by this instruction:

a. NWSCC 4082/2, T&ME Repair Record.

b. NAVSURFWARCENDIV 4355/73, Instrument Out-of-Tolerance Notice (OTN).

c. NWSCC 4734/2, Equipment Restricted From Use (Tag).

d. NWSCC 4855/16, Calibration Delivery Receipt.

e. NAVSURFWARCENDIV 10510/10, Instrument Status Report

f. NAVSURFWARCENDIV 10510/12, No Calibration Required Request.

W. E. SHOTTS

Distribution:
List A1-1, A3, C1
AFGE 1415
FOP 158
051 Files

CALIBRATION PROGRAM

RECORD OF CHANGES

Revision	Date	Description

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CHAPTER 1

INTRODUCTION

1.1 **REQUIREMENTS**. Calibration is mandatory for Test and Measurement Equipment (TME) used to make, record or generate quantitative measurements or data in the development, production, inspection or testing of material. Calibration is performed to the maximum intervals and requirements as described in NAVSEA OD 45845. Only those standards listed on the Calibration Standards Allowance Requirements (CALSTAR) or those allowed by the Calibration Manager may be used for calibrating TME.

1.2 **REFERENCE DOCUMENTS**

a. NAVSEA OD 45842, Calibration Facility Requirements for Shorebased Navy Calibration Laboratories.

b. NAVSEA OD 45845, Metrology Requirements List (METRL).

c. NAVSEAINST 4734.1, NAVSEA Metrology and Calibration Program

d. NAVSEA ST700-AM-GYD-010/METCAL, Metrology and Calibration (METCAL) Laboratory Requirements and Certification Guide.

1.3 **DEFINITIONS**. The following definitions are applicable to the performance of the Division's Calibration Program:

a. Ancillary Test Equipment. Equipment used in support of an inspection, measurement, or test that is not used to gather data. Ancillary test equipment does not require calibration prior to use. Inputs/outputs of ancillary test equipment will be monitored by calibrated equipment. Examples of ancillary test equipment include (but are not limited to) power supplies and amplifiers.

b. Calibration. The comparison of a measurement system or device of unverified accuracy with a measurement system of known and greater accuracy to detect and correct any deviation from required performance specifications of the unverified measurement system or device.

c. Calibration Intervals. Calibration intervals for all portable and installed TME are in accordance with NAVSEA OD 45845 with their approved calibration procedure. The information contained in NAVSEA OD 45845 is used as a ready reference to determine authorized calibration intervals, approved calibration procedures and related technical data. Technical changes or

additions may be applied to NAVSEA OD 45845 and the Instrument Calibration Procedures using NAVWPNSTA Seal Beach Form 13990/3, Calibration Problem Report. Calibration Laboratory personnel are responsible for submitting Calibration Problem Reports when a problem is detected. The Calibration Laboratory reserves the authority to EXTEND calibration due dates of specific TME as dictated by unusual circumstances and on a case-by-case basis.

d. Calibration Void Seal (Break-Out-Inspection (BOI)). Seals are attached to critical adjustment features of test equipment. The current calibration of the test equipment is void if a seal is broken.

e. Equipment Custodian. An individual appointed within an organization to act as a single point of contact for one or more pieces of test equipment.

f. Recall Report. A report that is generated by the Calibration Laboratory showing test equipment designated for recall. The recall report indicates items due for calibration, items that are due for calibration next month, and items that are overdue (delinquent) for calibration.

g. On-Site Calibration. Those TME which are not electrically or physically feasible to be transported to the Calibration Laboratory can be calibrated on-site.

h. S & C Number. An unique number assigned to equipment by the Calibration Laboratory. The number is imprinted on a tag or a bar code label assigned to the test equipment. The bar code may be affixed to the equipment, to a tag affixed to the equipment, or the case or container associated with the test equipment. **Note: A missing or illegible S & C Number does not void the calibration of the test equipment provided that the item is traceable through the serial number and the calibration records show the equipment calibration as current.**

i. Special Test Inspection Equipment. Directorates utilizing production jigs, fixtures, tooling masters, templates, patterns and other such devices for inspection purposes are responsible for ensuring these devices are proven for accuracy prior to use. These devices are assigned an S&C number and included in the calibration program. Directorates requiring certification of special test inspection equipment will be responsible for notifying the Calibration Laboratory of the requirement and provide all required documentation to adequately perform the certification. The Calibration Laboratory will perform certification of the device and enter it into the calibration recall system for periodic re-certification.

j. Stamp. An imprint on the calibration label or tag to authenticate the calibration and to identify the technician and the laboratory who performed the calibration.

k. Test and Measurement Equipment (TME). Any system or device used to evaluate the operational condition of an end item or subsystem thereof, or to identify and/or isolate any actual or potential malfunction. TME includes diagnostic and prognostic equipment, semiautomatic and automatic test equipment (with software), and calibration test or measurement equipment. All of the measuring instruments, measurement standards, and reference materials, auxiliary apparatus, and software which are integral to an instrument that is necessary for the operation and calibration of the instrument. This term includes measuring equipment used in the course of testing and inspection, as well as that used in calibration. This term is used interchangeably with "test equipment."

l. Test Equipment. Gages, instruments, inspection tools, fixtures, transducers, and measuring, monitoring, analysis, or diagnostic equipment used to measure static or transient phenomena to determine the characteristics or conformance to specifications of an article, material, system, process, or environment. This term is used interchangeably with "TME."

m. Test Software. Software which controls an automated test system or conditions a data signal from the point of measurement to the end point of use.

n. Traceability. The property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of calibrations.

CHAPTER 2

RESPONSIBILITIES

2.1 **CALIBRATION LABORATORY AND REPAIR BRANCH.** The Calibration Laboratory and Repair Branch (Code 6034), hereafter called Calibration Laboratory, is responsible for:

a. Managing, operating and maintaining the Division's Navy Calibration Laboratory in accordance with NAVSEAINST 4734.1, NAVSEA OD 45842 and NAVSEA ST700-AM-GYD-010/METCAL.

b. Providing metrology and calibration services for Division activities' TME and commensurate with their requirements.

c. Providing calibration services for other DOD activities as directed.

d. Performing initial calibration and acceptance testing of TME purchased by Division activities prior to acceptance for payment, as required.

e. Maintaining calibration/inventory/historical records for active and inactive TME for which the Division is accountable.

f. Performing operational re-verification tests and/or calibration of TME following repair actions.

g. Maintaining and issuing calibration recall notification for TME serviced by the Calibration Laboratory.

h. Providing calibration history data to the Metrology Science Directorate (MSD), Pomona, California for analysis in accordance with NAVSEAINST 4734.1.

i. Properly labeling/tagging TME serviced by the Calibration Laboratory as appropriate.

j. Notifying using activities of any out-of-tolerance condition detected during the calibration process.

k. Providing pickup/delivery services for TME calibrated in the Calibration Laboratory.

l. Maintaining a current file of all approved Navy calibration procedures, manufacturers manuals and gage drawings as required to support calibration efforts.

m. Developing and/or assisting in the development of local calibration procedures.

n. Establishing and assigning calibration intervals for TME in accordance with the guidelines of NAVSEAINST 4734.1 and NAVSEA OD 45845.

o. Certifying and providing Navy Special Interface Gages in support of Division requirements.

p. Providing training, advice and guidance on the use and care of gages as requested.

q. Developing and maintaining procedures describing how tasks associated with the Calibration Program are accomplished within the Calibration Laboratory.

r. Maintaining formal training plans and records for calibration technicians. Training may consist of either on-the-job-training and/or classroom/formal training.

2.2 **DIRECTORATES and TENANT ACTIVITIES**. Directorates and tenant activities are responsible for:

a. Appointing equipment custodians and alternates for calibration matters. Custodians may be appointed for each sub-organizational unit or building, as deemed necessary. A list of equipment custodians and alternates is to be sent to the Calibration Laboratory.

b. Verifying that all TME are properly labeled and those requiring calibration have been calibrated and placed in the calibration recall system.

c. Establishing with the Calibration Laboratory mutually agreeable secure areas for equipment pick-up and delivery.

d. Ensuring that equipment custodians or alternates are available at the pickup area as required.

e. Providing and maintaining Calibration Delivery Receipt (Appendix A) records of equipment transported to and from the Calibration Laboratory.

f. Notifying the Calibration Laboratory via "Instrument Status Report" form (Appendix B) of any changes in status of equipment.

g. Identifying and removing from use (impound) TME found to be defective or not in a valid and current calibration status.

h. Maintaining history usage records of individual items of TME used to make quantitative measurements or final acceptance inspection of any product.

i. Responding to Instrument Out-of-Tolerance Notice (OTN) memorandums.

j. Notifying the Calibration Laboratory of any foreseeable calibration support requirements of a new, unusual or unique nature.

k. Providing technical assistance to the Calibration Laboratory in support of system calibration services (Chapter 9).

l. Ensuring all contract and locally repaired TME is submitted to the Calibration Laboratory for re-verification/ calibration, as required.

m. Assuring that TME found to be defective is tagged with an "Inactive" or "Rejected" label and segregated from active TME.

n. Submitting all newly acquired TME to the Calibration Laboratory for assignment of an S & C number and entry into the calibration recall system.

2.3 **EQUIPMENT CUSTODIANS.** Equipment custodians are responsible for:

a. Serving as point of contact for questions concerning TME under their cognizance.

b. Ensuring that TME due for calibration is placed in the designated pickup area as appropriate.

c. Completing and/or signing the "Calibration Delivery Receipt" form (Appendix A) for TME delivered/received to/from the Calibration Laboratory.

d. Being available at designated pickup and delivery points for submission/receipt of equipment serviced by the Calibration Laboratory.

e. Submitting "Instrument Status Report" form (Appendix B) for TME under their cognizance, as required.

f. Verifying that TME directly exposed to combustibles, oxidizers, poisons, toxics and other sources of dangerous contamination are thoroughly examined for contaminates and decontaminated as required prior to submission for calibration.

g. Reconciling equipment delinquent for calibration with the Calibration Laboratory manager.

2.4 **TME USERS**. Users of TME are responsible for assuring that work is being conducted using equipment that have valid and current calibration labels.

CHAPTER 3

CALIBRATION PROGRAM PROCEDURES

3.1 **CALIBRATION SERVICES**. The Calibration Laboratory provides the following calibration services commensurate with Division requirements:

a. Laboratory calibration of Electrical/Electronic and Physical/Dimensional TME, and considered to be transportable to and from the calibration laboratory.

b. On-site system calibration services for automated, semi-automated TME and instruments comprising a system which are not feasible for delivery to the calibration laboratory.

c. On-site calibration services for certain gages, scales, weights and other TME as appropriate and approved by the Calibration Laboratory.

3.2 **PROCESSING OF TME FOR CALIBRATION**. The procedure for processing TME for calibration is shown in Figure 1 and described below:

3.2.1 Calibration Recall Notices. Monthly, the Calibration Laboratory sends a recall list to each equipment custodian identifying the equipment due for calibration and the date on which it is due. Equipment custodians review the recall report to identify any special requirements for the listed TME and to verify the equipment status. The equipment custodian notifies the Calibration Laboratory of a change in status of the equipment via "Instrument Status Report" form (Appendix B).

3.2.2 User Calibration. User Calibration is a category of TME that in order for it to function properly, due to its design, function or operational characteristics, requires calibration prior to each use or other specified frequency. Calibration of such equipment and maintenance of calibration history records is the responsibility of the using organization. Only those items designated by the Calibration Laboratory will be calibrated by the user. **Note: "User Calibration" is not the same authority and responsibility as "User Certification".**

3.2.3 External Calibration Services. When calibration requirements are beyond the capabilities of the local Calibration Laboratory, calibration services are obtained from external sources. These sources shall be recognized as an authorized calibration facility. Agreements with these external calibration facilities shall include the requirement of calibration data

being provided for recording in the local calibration data management system.

3.2.4 Priority Calibration Services. When circumstances warrant, equipment custodians may request priority calibration services via "Instrument Status Report" form (Appendix B). To minimize the impact of such services, the organizational manager will review these needs and if approved, so designate by their signature on the request.

3.2.5 On-Site Calibration. Equipment custodian determines if equipment is to be calibrated on-site. If so, the Calibration Laboratory is contacted to schedule a time for the performance of the calibration.

3.2.6 Assemble Equipment. For equipment calibrated in the Calibration Laboratory facilities, the equipment custodian gathers the TME due for calibration in the designated pickup area.

3.2.7 Calibration Delivery/Receipt Records. Equipment custodians are responsible for providing and maintaining Calibration Delivery Receipt records for each TME item transferred to and from the Calibration Laboratory. Equipment custodians identify on a "Calibration Delivery Receipt" form (Appendix A) the TME transferred to and from the Calibration Laboratory. For each piece of TME, the S&C number or drawing number is listed on the form and the form is validated by signature of the individual receiving the TME.

3.2.8 Initial Inspection/Test. The Calibration Laboratory will initially perform a test to determine the current state of equipment received for calibration. If equipment fails the initial tests, the following actions are taken:

a. If an out-of-tolerance condition is discovered, the procedure documented in Chapter 5 is implemented.

b. If failure is due to equipment problems, the equipment may be repaired locally or shipped to a vendor for repair. To assure that calibration/re-verification is performed on TME repaired, the following procedure will be followed:

(1) Organizations performing repair of TME normally calibrated by the Calibration Laboratory are responsible for submitting the item for calibration/re-verification when the repair is completed. The "T&ME Repair Record" form (Appendix C) shall be completed and submitted with the repaired equipment.

(2) Circumstances may exist where TME deployed off-site and requiring repair may for economic or technical reasons, be repaired by the facility to which assigned. In these

cases, a qualified calibration facility should be utilized for the calibration/re-verification action. The Calibration Laboratory will provide assistance in the selection of such a facility. In all cases, repairs of this category must be coordinated with and data provided to the Calibration Laboratory in order to update status and history records.

(3) TME designated as "Calibration Not Required" is excluded from re-verification following repair actions.

3.2.9 Calibration Process. Equipment is calibrated by Calibration Laboratory technicians using calibration procedures. After the completion of the calibration process, the appropriate calibration label is attached to the TME (see Chapter 4), a calibration interval is assigned, appropriate information is entered into the Calibration Data Management System (see Chapter 6), and equipment returned to the equipment custodian using the "Calibration Delivery Receipt" form (Appendix A). Some TME require special processing (see Chapter 7).

3.2.10 Test Software. Where test software is used as a form of inspection, it shall be proven to be capable of verifying the acceptability of product prior to the test software's release for use during production. Test software configuration is to be verified prior to each use. Note: Test software used successfully prior to the release of this instruction is deemed to be verified by demonstration.

3.3 **PROCESSING OF NEW EQUIPMENT.** The processing of new equipment (equipment not previously entered into the calibration recall system) is described in Figure 1.

3.4 **RECALL DELINQUENCY.** TME is placed on a delinquency list if it has not been received in the Calibration Laboratory within 30 days of the calibration due date. The equipment custodian should make every effort to reconcile the delinquency with the Calibration Laboratory manager.

FIGURE 1 CALIBRATION PROCESS FLOW DIAGRAM (MONTHLY RECALL)

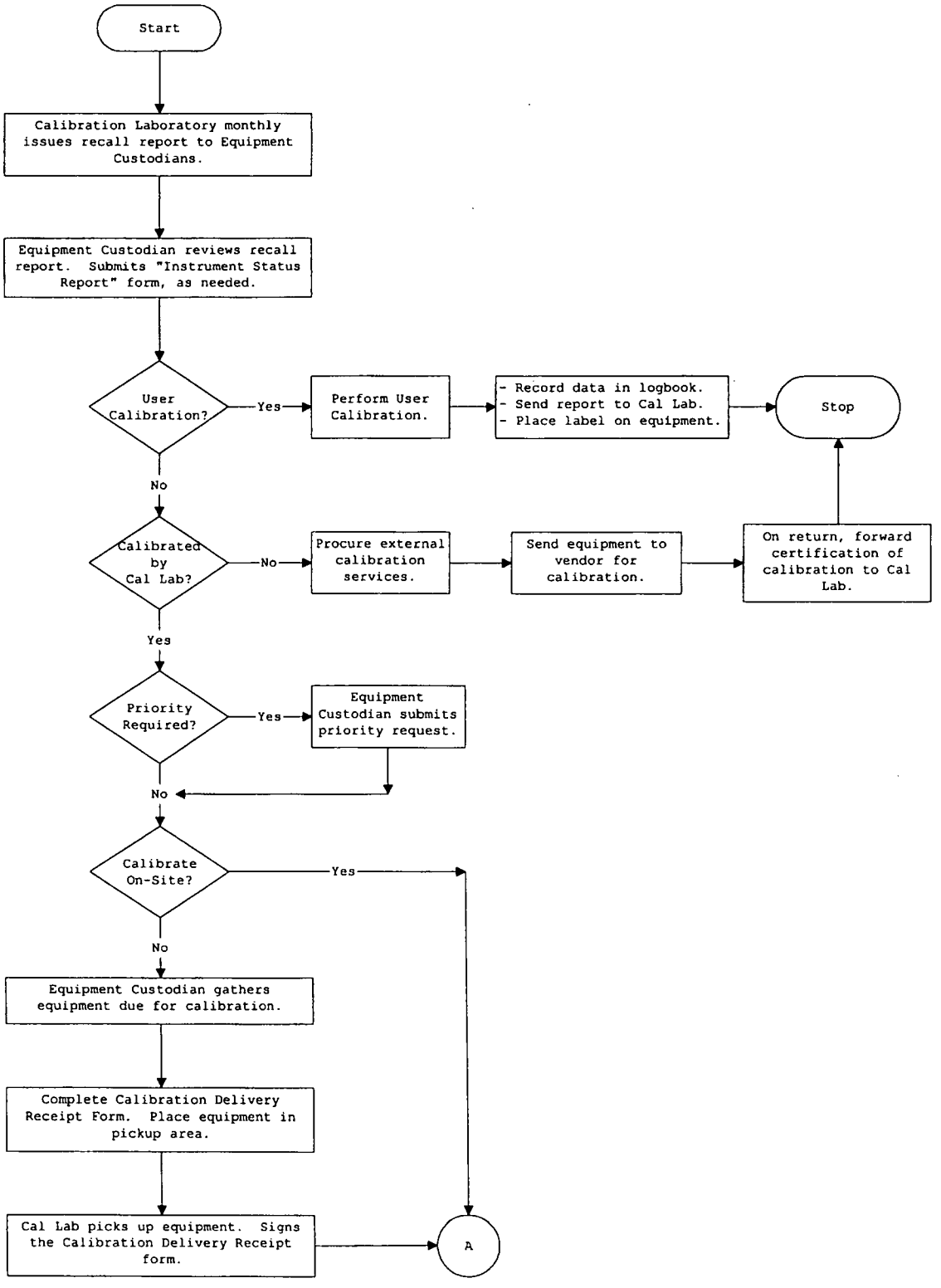


FIGURE 1 CALIBRATION PROCESS FLOW DIAGRAM (Continued) (CALIBRATION PROCESS)

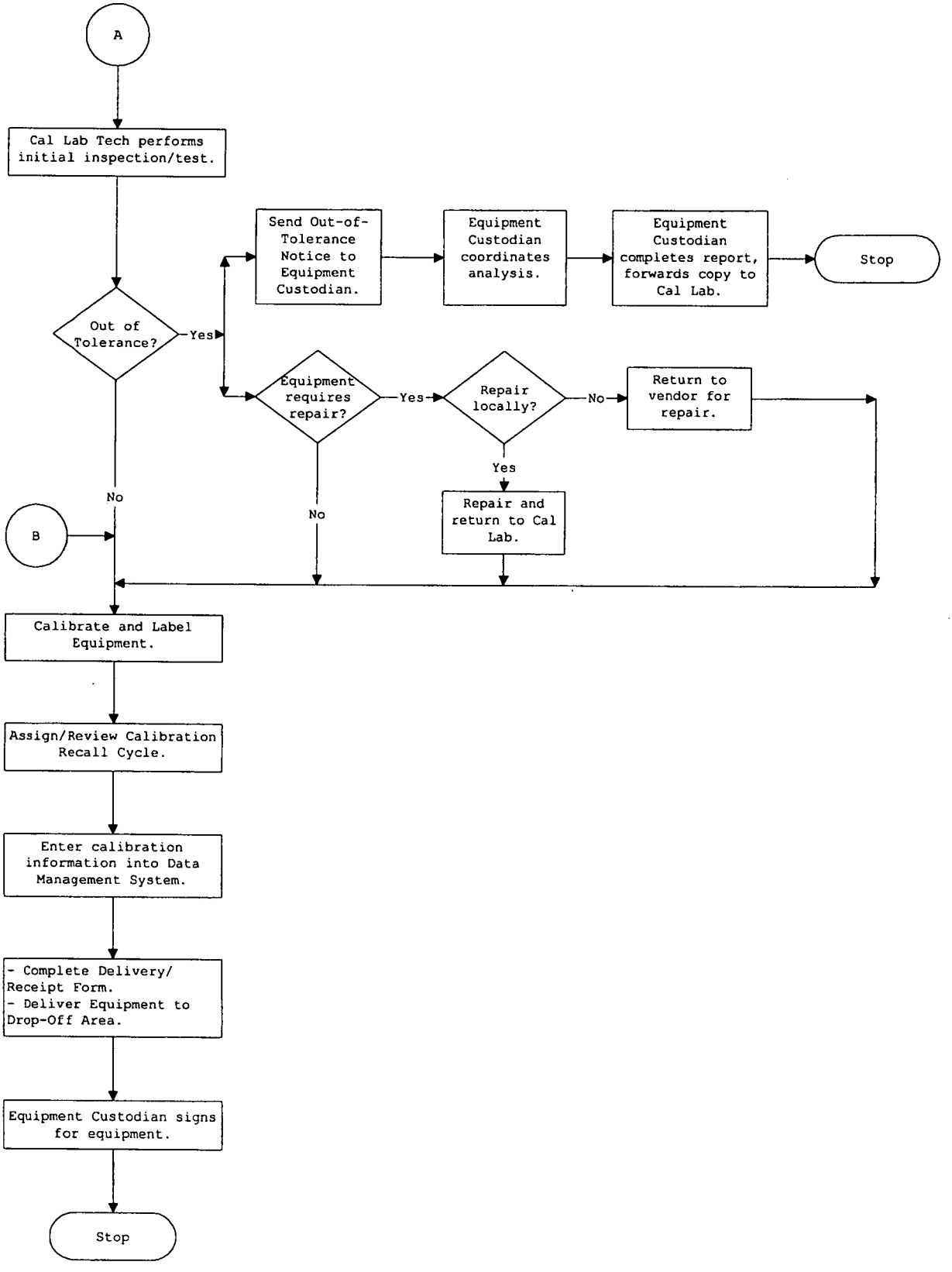
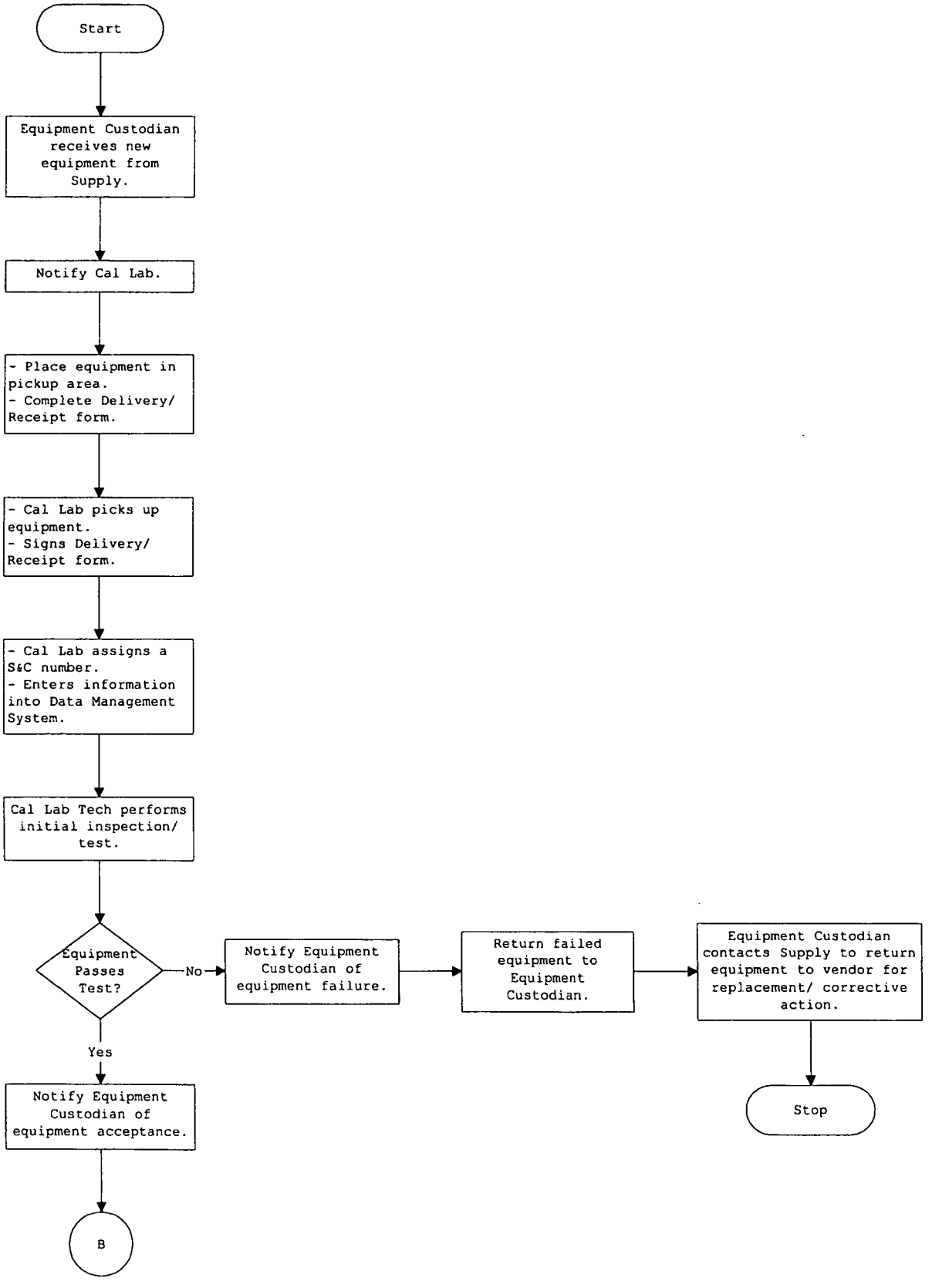


FIGURE 1 CALIBRATION PROCESS FLOW DIAGRAM (Continued) (NEW EQUIPMENT PROCESS)



CHAPTER 4

CALIBRATION STATUS AND LABELS

4.1 **INTRODUCTION.** Calibration labels (or tags) are attached to all TME and are used to indicate the calibration status. The Calibration Laboratory is the only Division activity authorized to affix calibration labels or tags to TME. Further, it is the responsibility of the Calibration Laboratory to ensure that such labels (and tags) are properly controlled, maintained and appropriately applied to TME at time of calibration/verification. **Note: A missing or illegible label does not void the calibration of the test equipment provided the calibration records show the equipment calibration as current.**

4.2 **APPLICATION GUIDANCE.** The following guidelines apply to completion and location of calibration labels placed on TME:

a. Calibration labels will be completed showing calibration status, date calibrated, due date and by whom calibrated (Calibration Laboratory code).

b. Whenever possible, all calibration labels/tags will be affixed using as large a label or tag as appropriate in a position visible to persons facing the instrument and in the normal viewing plane of the instrument.

c. It is sometimes impossible to locate calibration labels on instruments where they will not be destroyed during use. In such cases, the proof of calibration will be placed on the box containing the instrument or on a tag/record card accompanying the instrument. This tag/card will be readily available at all times and will identify the TME to which it applies.

d. If the tag/record card displaying the calibration label becomes lost, destroyed or illegible, the Calibration Laboratory should be notified. A replacement will be issued when current calibration status is verified by review of calibration history data.

4.3 **CALIBRATION LABELS.** Calibration labels and tags applicable to the Division's Calibration Program are described as follows:

a. CALIBRATED. This label (black lettering, white background), indicates that the equipment is within its applicable tolerance on all parameters. If there are any qualifying conditions for using the instrument, one of the other labels described below is used. The label shows the calibration status, date calibrated, due date and the technician code who performed the calibration.

b. SPECIAL CALIBRATION. This label (black lettering, yellow background), is used whenever there is some unusual or special condition in the calibration which must be drawn to the attention of the user. The special condition may be deviations from usual calibration tolerances, multiple calibration intervals, or a requirement of in-place calibration. The special condition requiring the use of this label must be described directly on label or tag. The following provides a brief description of special conditions:

(1) Deviation from Specifications (Limited Calibration). In cases where the user does not require full equipment capability, calibration is performed to reduced tolerances or covers less than all ranges and parameters. This approach is often used when the equipment does not meet full calibration tolerances on certain ranges or parameters but can still meet user requirements. On the other hand, the special calibration may be for higher accuracy than usual on a short-term basis upon the specific request of the user.

(2) Multiple Calibration Intervals. Some systems have components which require calibration less frequently than the rest of the equipment. For example, the attenuator in a signal generator may require calibration every 12 months; whereas, the rest of the equipment parameters should be calibrated every four months. Since the attenuator calibration is time-consuming and may require unavailable standards, using the multiple interval approach can save considerable work-hours. When a specific piece of equipment has been designated for multiple calibration intervals, such information is provided in the applicable calibration procedure. The "Special Calibration" label or tag is annotated with "Multiple Interval" and the type of calibration performed is indicated. The calibration due date reflects the next partial or complete calibration.

(3) Calibrated In-Place. Some equipment should be calibrated in-place. Annotating the "Special Calibration" label or tag will alert both the user and calibration technician that the equipment should not be removed, but should be calibrated in-place.

c. CALIBRATION STANDARD. This label (black lettering, blue background) is used to identify Navy Metrology and Calibration (METCAL) Program calibration standards. The "Calibration Standard" label will be affixed to all equipment specifically designated for use by Navy standards and calibration activities.

d. USER CALIBRATION. This label (black lettering, white background) is affixed when the calibration is performed by the user; however, this label is not replaced at each calibration. When the label is first affixed to the instrument, it is annotated as the appropriate calibration interval. Some TME

reliability or operating characteristics must be established prior to use or at very short calibration intervals. These can be calibrated by the user as opposed to referring the instrument to a calibration facility. For example, some instruments, such as hardness testers and densitometers are provided with standards and should be calibrated with every use or very frequently. Some instruments, such as oscillographic recorders, may require calibration before, during and after use. Still other instruments are calibrated as part of check-out procedures performed daily or weekly and recorded in maintenance logs. The requirement for calibration by the user and the calibration interval is indicated in the Metrology Requirements Lists (METRL). Records of calibrations performed should be by normal maintenance practices, e.g., maintenance log and Maintenance Action Form.

e. REJECTED. In the event an instrument fails to meet the acceptance criteria during calibration and cannot be adequately adjusted, a "Rejected" label (black lettering, red background) is placed on the instrument and all other servicing labels removed. In addition to the "Rejected" label. A "Rejected" tag giving the reason for rejection and other information as required is affixed to the instrument. The "Rejected" label and tag remains on the instrument until it is repaired or re-calibrated. The instrument will be segregated and not used while bearing a "Rejected" label.

f. BREAK-OUT-INSPECTION SEALS. Break-Out-Inspection (BOI) seals consist of labels (black lettering, white background) bearing the words "CALIBRATION VOID IF SEAL BROKEN", sealing compounds, or lead seals, as appropriate. BOI's are placed over readily accessible (usually exterior) adjustments to prevent tampering that could affect calibration. They are also used to prevent removal and/or interchange of plug-ins, modules, and subassemblies when such removal would affect the calibration. TME bearing a broken BOI should be considered as "not calibrated" and must be calibrated/re-certified prior to use.

g. INACTIVE. TME having no foreseen requirements for 30 or more days beyond its calibration due date may be removed from calibration recall and affixed with an "Inactive" label (green lettering, white background). Equipment in this category will not be recalled until the user determines that it is again needed. The following procedure will be used to place equipment in an inactive status:

(1) Using activity will notify the Calibration Laboratory via "Instrument Status Report" form (Appendix B) that an item is to be placed in an inactive status.

(2) The Calibration Laboratory will change calibration records accordingly and have an "Inactive" label placed on the item. The owning activity will then separate the item from

active calibrated equipment by placing it in an impound area. If this is not practical, it must be tagged as restricted from use.

(3) When the instrument is again needed, the owning activity will place it in the calibration pickup area and initiate a status change request via "Instrument Status Report/Priority Request" form. The item will then be re-entered into the calibration recall system.

h. EQUIPMENT RESTRICTED FROM USE. TME that does not bear a valid and current calibration label must be restricted from use. Such equipment must be labeled with a completed "Equipment Restricted From Use" tag (Appendix D) and physically impounded (if practical) when any of the following conditions exist:

(1) Equipment is unlabeled.

(2) Equipment that has passed its calibration due date.

(3) Equipment is labeled as inactive.

(4) Equipment has a broken BOI seal or sealing compound.

(5) Equipment has failed in operation.

(6) Equipment is suspected of being outside of its performance limits.

i. CALIBRATION NOT REQUIRED. This label (orange lettering, white background) is to remain affixed to equipment designated as "Calibration Not Required" (CNR). Equipment qualifying as CNR fit in two separate groups: (1) calibration not required by METRL, and (2) calibration not required due to usage. The following procedure is used for placing equipment in a CNR status:

(1) Equipment not requiring calibration in accordance with METRL will be labeled as such when entered into the calibration recall system by the Calibration Laboratory.

(2) Equipment used for indication purposes only, that would otherwise require calibration, may be placed in CNR status when requested by the user and approved by the Calibration Laboratory.

(3) Request for CNR will be via the "No Calibration Required Request" form (Appendix E) and must be completed and signed by the organization's manager. The Calibration Laboratory will review the usage conditions of the equipment for which the CNR is requested and upon concurrence place the equipment in a CNR status and apply the appropriate label. The using activity is responsible for proper utilization of TME placed in a CNR

status. The following criteria is to be used as guidance for placing equipment in a "Calibration Not Required" status:

(a) The equipment is not used to make quantitative measurements or provide quantified outputs.

(b) The device is "fail-safe" in that operation beyond specified tolerances will be apparent to the user.

(c) Measurement and stimulus circuits are monitored during use by calibrated equipment, or are dependent on an external known or calibrated sources for performance within required limits.

CHAPTER 5

OUT-OF-TOLERANCE TME

5.1 **NOTIFICATION.** Using activities are notified by the Calibration Laboratory when an out-of-tolerance condition is discovered during the calibration process that could adversely impact TME measuring accuracies, quality of performance or integrity of the unit. An "Instrument Out-of-Tolerance Notice (OTN)" form (Appendix F) is prepared by the Calibration Laboratory when any of the following conditions exist:

a. TME under calibration fails any of the measurement parameters tested.

b. TME received for calibration has a broken Break-Out-Inspection (BOI) seal or sealing compound as required on certain gages.

c. TME is received from a local repair activity without a completed "TME Repair Record" form (Appendix C).

5.2 **OTN DISTRIBUTION/ACTION.** Copies of the OTN are sent by the Calibration Laboratory to the cognizant equipment custodian. It is the responsibility of the using activity to respond to OTNs by conducting a review and analysis of the reported out of tolerance condition in terms of adverse impact to product quality and relative to the TME application. Results of this analysis and action taken is to be reported to the Calibration Laboratory for record purposes.

CHAPTER 6

CALIBRATION RECORDS

6.1 **CALIBRATION DATA MANAGEMENT.** The Calibration Laboratory operates, manages and maintains an automated, calibration recall system for the Division. This system is used to maintain calibration/history/inventory, and to the extent possible, repair information pertaining to TME for which the Division is accountable. Included in the system is all TME used to make, record or generate quantitative measurements or data in the development, production, inspection or testing of material. All active and inactive TME, calibration standards and standard support equipment is included in the system. In addition, history records are maintained for all calibrations performed by off-site activities when such calibration is in support of TME under the cognizance of the Division.

CHAPTER 7

SPECIAL TME

7.1 NAVY SPECIAL INTERFACE GAGES

7.1.1 The Navy Gage and Standards Center (NGSC), Naval Weapons Station, Seal Beach Detachment, Pomona, California, is designated as the technical authority for combat systems Special Interface Gage requirements within NAVSEASYSCOM. This responsibility includes technical and administrative design, configuration management, procurement, maintenance, modification, inventory control, issue and certification support. NAVSURFWARCENDIV Crane Calibration Laboratory has been delegated authority by NGSC, Pomona to certify, modify and maintain Navy Special Interface Gages (NSIG) required by the Division and used in support of conventional ammunition production. Specifically excluded from this authority is certification/re-certification of NSIG required in support of missile programs.

7.1.2 Activities having requirements for NSIG under cognizance of NGSC, Pomona should telephone the requirement to the Calibration Laboratory by gage drawing number or dimensional characteristic to be gauged. The Calibration Laboratory will initiate action to obtain, certify and issue those gages.

7.2 **SPECIAL TEST INSPECTION EQUIPMENT**. Division activities utilizing production jigs, fixtures, tooling masters, templates, patterns and other such devices for inspection purposes, are responsible for ensuring that these devices are proven for accuracy prior to use. Further, such devices are to be included in the calibration recall system. Activities requiring certification of special test inspection equipment will be responsible for notifying the Calibration Laboratory of the requirement and providing documentation required to adequately perform the certification. The Calibration Laboratory will perform certification of the device and enter it into the calibration recall system for periodic re-certification.

CHAPTER 8

USER CERTIFICATION PROCEDURES

8.1 **INTRODUCTION.** This procedure describes the request and approval method, conditions and requirements of User Certification authority as delegated by Calibration Laboratory to individual users. The Calibration Laboratory recognizes that conditions may exist where the calibration of certain TME would be more practical and economical when performed by the user. These conditions may exist or result from the uniqueness of the equipment, special application or system complexity and configuration.

8.2 **REQUEST FOR USER CERTIFICATION.** Division users of TME who feel that their equipment may qualify for User Certification authority under any of the categories mentioned above are responsible for requesting that authority, via memorandum, to Calibration Laboratory. The Calibration Laboratory will review the request in terms of equipment usage, calibration support equipment requirements, calibration procedures and training requirements. Based on the results of the review, the Calibration Laboratory will then determine if User Certification is appropriate.

8.3 **USER CERTIFICATION REQUIREMENTS.** Where User Certification is approved, specific conditional requirements pertaining to personnel qualifications, equipment control and performance monitoring will be defined in writing by the Calibration Laboratory. These requirements may include, but not be limited to, the following:

a. Documented training (formal/OJT) and/or experience.

b. Scope of authority.

c. Testing and examination of personnel.

d. Instructions/procedures/guidelines to be followed.

e. Random monitoring and/or audits of certified personnel.

f. Reports and data pertaining to calibrations performed.

g. Re-certification procedures.

h. Conditions under which certification may be revoked.

i. Period of certification.

CHAPTER 9

SYSTEM CALIBRATION PROCEDURES

9.1 **INTRODUCTION.** This procedure describes the request and review method, conditions and responsibilities for acquiring system calibration services and/or delegation of such authority to the user by the Calibration Laboratory. System calibration services are provided by the Calibration Laboratory for equipment belonging to the Division when so requested by the user and approved by the Calibration Laboratory. Normally, systems are calibrated on-site. Conditions may exist where on-site calibration is not possible due to calibration accuracy ratio, environmental conditions and limited portability of calibration standards. In such cases, these systems/system components will require laboratory calibration support.

9.2 **SYSTEM CALIBRATION CONDITIONS.** TME shall be considered as a "System" and calibrated as such when any of the following conditions exist:

a. Items defined as Automated Test Equipment (ATE) or Semi-Automatic Test Equipment by NAVSEAINST 4734.1.

b. Any array of instrumentation that is interfaced through adapters, cables, dividers, switches, amplifiers, or other means for the purpose of monitoring, measuring or controlling any test parameter(s) and where removal of any single unit would render the calibration of the unit or system invalid.

c. An interfaced configuration of multiple TME dedicated to and used in support of specific long term test. Under normal usage conditions, TME comprising such a system would be calibrated as "stand alone" items.

9.3 **SYSTEM CALIBRATION REQUESTS.** Division organizations using TME that they feel may warrant system calibration are responsible for notifying the Calibration Laboratory of their requirement. Organizations shall send written notification to the Calibration Laboratory requesting system calibration services along with applicable drawings, schematics, test and facility requirements necessary to determine feasibility of system calibration. The Calibration Laboratory will review the request and determine the appropriateness and feasibility of system calibration based on the above conditions.

9.4 **USER CERTIFICATION.** Normally, system calibration is performed by Calibration Laboratory personnel, however, conditions may exist where it is more appropriate for the user to perform calibration of specific systems. This may be due to the complexity or uniqueness of the system, special training or

certification requirements and other extenuating circumstances. Where User Certification is deemed appropriate, certification of personnel is to be accomplished in accordance with the User Certification Procedures (Chapter 8).

9.5 **SYSTEM CALIBRATION GUIDELINES.** Where system calibration services are appropriate and agreed upon, the following guidelines apply:

a. The using activity shall provide:

(1) A calibration procedure and/or assistance in development of a calibration procedure, as required.

(2) A copy of the system calibration software, as appropriate, to be retained by the Calibration Laboratory and used in the calibration/re-verification process.

(3) Assistance in the operation and calibration of the system as required and mutually agreed to the Calibration Laboratory.

(4) A complete inventory of instrumentation comprising the system, listed by manufacture, model number and serial number.

(5) Proper maintenance of records as required by the Calibration Laboratory to control instruments assigned to the system.

(6) A technical point of contact to address questions and/or problems associated with the system.

b. A calibration history log book shall be maintained by the user for each system under their cognizance. As a minimum, this log shall contain:

(1) Manufacture, model and serial numbers of each item comprising the system.

(2) Chronological record of broken BOI seals and supporting reasons.

(3) Record of all repair actions and date performed.

(4) System re-verification actions and dates.

(5) Calibration date and due date.

c. TME assigned to a system may be removed to serve as stand alone equipment provided:

(1) The instrument has been calibrated by the Calibration Laboratory and bears a valid and current calibration label.

(2) All necessary log entries are made indicating the instrument has been removed from the system.

(3) Re-verification procedures have been executed prior to placing the system back in an active status.

d. If there is an equipment failure, TME of the same manufacture and model number may be placed in the system to minimize downtime or aid in failure analysis. Re-verification of the system is required after such action and appropriate entries made in the system log.

e. The Calibration Laboratory is to be notified of any change or alterations to equipment comprising a system or if there are any modifications and/or changes to the calibration software.

f. The Calibration Laboratory will maintain complete calibration history information for the system in the Division calibration recall system.

g. Calibration recall cycles for all ATE and systems will be established by the Calibration Laboratory in accordance with NAVSEA OD 45845 and commensurate with the using activities requirements.

APPENDIX A

CALIBRATION DELIVERY RECEIPT FORM

CALIBRATION DELIVERY RECEIPT
NWSCC 4855/16 (REV. 10/86)

DATE	CUSTODIAN

ITEM	EQUIPMENT DESCRIPTION
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21.	
22.	
23.	
24.	
25.	

RECEIVED BY

APPENDIX B

INSTRUMENT STATUS REPORT FORM

INSTRUMENT STATUS REPORT
NAVSURFWARCENDIV 10510/10 (Rev. 5-99)

FROM	TO
CODE _____ BUILDING _____	CODE _____ BUILDING _____

STATUS CHANGE

Change the following to the status indicated:

S/C NO.	LEGEND NO. *

LEGEND*

*USE NUMBERS LISTED BELOW FOR LEGEND COLUMN FILL-IN.

1. EQUIPMENT TRANSFERRED TO CUSTODIAN CODE _____ BLDG. _____
2. EQUIPMENT TRANSFERRED OFF-CENTER.
3. EQUIPMENT RETURNED TO FACTORY.
4. EQUIPMENT IN "INACTIVE STATUS".
5. EQUIPMENT TO BE TRANSFERRED TO "USER CALIBRATION" STATUS.
6. EQUIPMENT SURVEYED OR SCRAPPED.
7. EQUIPMENT LOST.
8. OTHER _____

CUSTODIAN *(Signature)*	DATE

APPENDIX C

T&ME REPAIR RECORD FORM

T&ME REPAIR RECORD
NWSCC 4082/2 (12-83)

SECTION I

PREPARER	CODE	CUSTOMER	DATE	S&C/I.D. NO.

NOMENCLATURE	SYSTEM

MODEL	SERIAL NO.	MANUFACTURER

PROCEDURE NO.	CHARGE NO. *(If direct)*

STATE FIRST INDICATION OF TROUBLE/FAILURE	IDENTIFY PARAGRAPH/TABLE/STEP/ETC.

DESCRIBE TROUBLE/FAILURE

SECTION II

STATE PROBABLE CAUSE

DESCRIBE ACTION TAKEN AND DISPOSITION OF FAILED ITEMS *(Identify piece parts as: Name/Schematic Reference Symbol/PN/NSN/QTY.)*	PART NO.	PRICE
☐ SEE CONTINUATION SHEET ☐ TOTAL		

STATE RECOMMENDATION/REMARKS - DID ACTION CORRECT PROBLEM?

TIME REQUIRED *(Hours and minutes)*

REPAIR AUTHORIZED BY	DATE

RECALIBRATION RECOMMENDED ☐ YES ☐ NO

COMPLETED BY	DATE

APPENDIX D

EQUIPMENT RESTRICTED FROM USE FORM

EQUIPMENT RESTRICTED FROM USE
NWSCC 4734/2 (6-91)

DATE	S/C No.

PENDING

- ☐ CALIBRATION
- ☐ REPAIR
- ☐ MODIFICATION
- ☐ LOCAL FABRICATION
- ☐ AUTHORITY FOR USE

APPENDIX E

NO CALIBRATION REQUIRED REQUEST FORM

NO CALIBRATION REQUIRED REQUEST
NAVSURFWARCENDIV 10510/12 (Rev. 5-99)

INSTRUCTIONS: A No Calibration Required (NCR) request must be completed on each item of Test Measurement Equipment (TME) for which NCR status is desired. Each request must be signed by the appropriate branch or project manager who has authority to determine project calibration support requirements. NCR should be requested only for a device that is in use and is normally calibrated but under current usage conditions does not require calibration. These conditions under which the device is currently being used must be described below. Upon receipt of this request, Code 6034 will update the Calibration Recall Database accordingly and return the request along with an NCR label to the requesting custodian. The NCR label will identify the S/C number of the instrument for which it was requested and is applicable only to that instrument. All existing calibration labels (except S/C number) must be removed before affixing the NCR label where clearly visible. The NCR label is valid only so long as the declared usage conditions do not change.

NOTE: It is the responsibility of the requesting and using activity to ensure the device is used in the manner described in this request and to notify Metrology/Calibration Branch, Code 6034 if usage conditions change. Failure to comply with this requirement is a violation of NAVSEA METCAL and Center calibration instructions. A signed copy of this request must be attached to the device or available in the immediate area where it is being used.

FROM: (ORG CODE) _____ BLDG: _____ CAL EQUIP CUSTODIAN CODE: _____

TO: _____ BLDG: _____

1. Request the following item be placed in a NCR due to usage status:

ITEM IDENTIFICATION:

S/C	MODEL No.	SERIAL No.	MANUFACTURER	NOMENCLATURE

2. NCR status for the above item is appropriate due to usage conditions as described in detail below. The device does not provide quantitative analysis data and use in a NCR status will not adversely affect product quality.

3. Describe in detail how the device is used:

_____ _____ _____

SIGNATURE OF REQUESTING OFFICIAL *(Must be Branch or Project Mgr.)* | DATE OF REQUEST | TELEPHONE NO.

(FOR CALIBRATION LAB USE ONLY)

Processed by _____ on _____

APPENDIX F

INSTRUMENT OUT OF TOLERANCE NOTICE (OTN) FORM

INSTRUMENT OUT-OF-TOLERANCE NOTICE (OTN)

OTN No.

FROM | TO

The following listed instruments belonging to your activity failed to meet manufacture's specifications during calibration.

SERVICE/ CALIBRATION NO.	MANUFACTURER	MODEL	SERIAL NO.	NOMENCLATURE

It is your responsibility to maintain a program to react to out-of-tolerance notices issued to you as required by NAVSURFWARCENDIV CRANEINST 4855.20. This includes analyzing the effect of equipment error on the acceptability of material or validity of data recorded. The attached data is the out-of-tolerance data taken during calibration and is forwarded to assist you in making this determination.

Results of your review must be forwarded to ☐ Code 6034 ☐ SMCCN-IOE within five working days of date of this memo.

SIGNATURE *(Section/Branch Manager)* | DATE

REVIEW RESULTS RESPONSE

FROM | TO ☐ CODE 6034, BLDG. 39 ☐ CODE SMCCN-IOE, BLDG. 13

COMMENTS

SIGNATURE *(Custodian)* | DATE

NAVSURFWARCENDIV 4355/73 (REV. 5/99)

NAVSURFWARCENDIVCRANE INSTRUCTION 4855.4

From: Commander

Subj: SHELF-LIFE MANAGEMENT

Ref: (a) NAVSURFWARCENDIVCRANEINST 4855.1, Quality System
(b) DoD 4140.27-M, Shelf-Life Management Manual
(c) Fed-Std-793, Depot Storage Standards

Encl: (1) Shelf-Life Management Program

1. Purpose. To document a program for identifying and controlling age sensitive material in accordance with the applicable requirements and guidelines of references (a) through (c).

2. Scope. This instruction applies to Naval Surface Warfare Center, Crane Division (NAVSURFWARCENDIV Crane) Crane, Indiana site organizations that procure, receive, store or use shelf-life materials.

3. Policy. The Division will procure, process and control shelf-life materials to assure they perform satisfactorily in service.

4. Action

a. The Supply Directorate shall establish appropriate controls to prevent deterioration, damage, degradation, or unauthorized issuance of shelf-life materials in storage in accordance with the applicable requirements of enclosure (1).

b. Directorates shall assure that shelf-life materials are properly controlled to ensure the integrity of their products and services in accordance with the applicable requirements of enclosure (1).

c. The Quality Management Office (Code QMO) shall maintain this instruction, and evaluate compliance and provide guidance on the implementation of the requirements in enclosure (1).

W. E. SHOTTS

Distribution:
List A1, A3, C1
AFGE 1415
FOP 158
051 FILES

SHELF-LIFE MANAGEMENT PROGRAM

RECORD OF CHANGES

Revision	Date	Description

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CHAPTER 1

INTRODUCTION

1.1 **PURPOSE**. This instruction documents the Shelf-Life Management Program at the Naval Surface Warfare Center, Crane Division (NAVSURFWARCENDIV Crane). The procedures contained in this instruction help accomplish the following:

a. Reduce the quantity of shelf-life materials declared excess due to age.

b. Reduce the generation of hazardous waste.

c. Reduce the costs associated with management of Type II (extendable) shelf-life material.

d. Reduce the costs associated with the replacement of expired shelf-life material within an end item.

1.2 **DISCUSSION**

a. Material whose serviceability changes over time is to be identified and controlled to prevent damage, deterioration, degradation, or unauthorized issue. Effective management of shelf-life material maintains the requisite level of stock availability and minimizes the risk of shelf-life material expiration prior to issue and use. Because shelf-life materials require special management with attendant additional costs, the designation of material as shelf-life is to be held to a minimum.

b. Shelf-life material is material that possesses deteriorative or unstable characteristics to the degree that a storage time period must be assigned to assure that it will perform satisfactorily. Shelf-life materials are categorized into two types:

(1) **Type I** - Material having a definite (non-extendable) period of shelf-life established by empirical and technical test data.

(2) **Type II** - Material assigned shelf-life time period that may be extended after the completion of prescribed inspection, test and/or restorative action.

CHAPTER 2

GENERAL REQUIREMENTS, PROCEDURES AND RESPONSIBILITIES

2.1 **SUPPLY DIRECTORATE.** The Supply Directorate is responsible for the procurement, receipt, storage, and management of shelf-life material within their stores; and the issuance of shelf-life material to using directorates.

2.1.1 **Acquisition** personnel, as part of the contract and procurement practice, shall:

a. Assure that purchase requisition records for shelf-life materials are properly completed, i.e. requisitions for shelf-life materials contain an inspection indicator code of "Y" indicating a requirement for receipt inspection.

b. Assure that purchase documents for shelf-life materials include a clause to require correct shelf-life information and marking; and a requirement that upon receipt, the material will have no less than 75% of its shelf-life remaining.

c. Assure that vendors and suppliers complete a Material Safety Data Sheet (MSDS) for shelf-life materials; maintain a copy on file and forward a copy to the requesting directorate.

d. Participate, when requested, as a member of Material Review Boards (MRBs) involving non-conforming shelf-life materials to assist in appropriate disposition.

2.1.2 **Material Management** personnel shall:

a. Inspect incoming shelf-life material for:

(1) Adequate remaining shelf-life.

(2) Transportation and handling damage.

(3) Signs of deterioration (e.g., leaky containers, evaporated contents, and broken seals).

b. Reject and return to vendor any shelf-life materials received with less than 75% of the assigned shelf-life remaining at time of receipt. National Stock Number (NSN) items are exempt from this requirement.

c. Prepare an appropriate discrepancy report for each instance of nonconforming material in accordance with applicable procedures. When the disposition is "Return to Vendor", prepare either a Report of Discrepancy or Product Quality Deficiency Report in accordance with applicable procedures.

d. Ensure shelf-life containers are affixed with a shelf-life label prior to storage or issue. If shelf-life containers are not affixed with a shelf-life label, prepare and apply labels containing the following minimum information:

(1) For Type I shelf-life material, use a red label and identify the disposal date.

(2) For Type II shelf-life material, use a white label and identify the manufacture/cure date, and the inspection/test interval.

e. Develop, maintain and update inventory records for Supply managed shelf-life materials.

f. Ensure shelf-life materials issued for use have not exceeded their assigned shelf-life and do not display undue indications of deterioration or damage.

g. Document and implement a purging and disposal process so that unserviceable and/or non-extendable expired shelf-life materials are purged from stock.

h. For material stored in the Automated Storage Retrieval System (ASRS), review the weekly report from the Mini Load Computer System (MLCS) of shelf-life material that has 30 days or less of shelf-life remaining to determine if the material has an extendable shelf-life code, and:

(1) If the material is extendable, determine if the material is still in serviceable condition. If so, extend the expiration date by adding a new expiration date to the record through the MLCS; prepare and apply a shelf-life extension label in accordance with paragraph 2.2f(4).

(2) If material is not extendable or is extendable but not serviceable, pull material from the location upon expiration and notify the Traffic item manager that the material has expired.

2.1.3 **Traffic** item managers, upon notification that material has expired, will, per applicable procedures, move the record to "H" condition (unserviceable), prepare a disposal document, and send material to the Defense Reutilization and Marketing Office for disposal.

2.2 **DIRECTORATES** shall:

a. Assign a shelf-life coordinator(s) at the appropriate level within the organization to perform functions pertaining to the management of shelf-life materials.

b. Establish and document controls necessary to assure that only serviceable materials are used in production and test operations.

c. Document actions taken to reclassify, exempt or otherwise modify requirements for managing shelf-life materials. Assure that tags, labels or other applicable markings reflect any changes made (see paragraph 2.2f(4)).

d. Monitor the Consolidated Hazardous Material Program (CHMP) to identify expired or expiring materials under Supply's cognizance which are needed to meet production requirements. If needed, request such material be issued. If material acceptability is in question (i.e., material has expired or will expire before use but is considered acceptable for a particular application), forward appropriate supporting documents (e.g., customer approvals or engineering dispositions) along with the request for material issuance.

e. Maintain information containing the nomenclature, shelf-life (SL) code and the expiration date of material currently on hand.

f. Inspect/test Type II (extendable) shelf-life material by practical, end-use related tests to determine if the material still meets the intended use. The shelf-life may be extended as long as the item performs satisfactorily for the end-user's needs. The following guidance and information is provided to aid in making shelf-life extension determinations:

(1) For NSN items, organizations may use the monthly DOD Quality Status Listing (QSL), available from Defense Logistics Agency (DLA), to identify which materials can be extended and for how long. The QSL contains the results of tests by Department of Defense and General Service Administration (GSA) laboratories to determine if type II shelf-life material may continue to be used.

(2) In-house inspections and tests are appropriate for most material. Guidance for GSA managed items is provided in FED-STD-793. Where descriptions of the specific inspections or tests required to extend the shelf-life of particular materials are not readily available, contact the Inventory Control Point (ICP) of the material for storage standards or use individual product specifications. Additional assistance can be obtained from Military Service and Agency Administrators for the DoD Shelf-Life Program listed in DoD 4140.27-M.

(3) For most Type II material, shelf-life extension tests are not complicated, do not require a laboratory, and can be done on the spot by the user. They are usually nothing more than visual checks for damage or deterioration. The criteria for visual inspection to extend shelf-life is as follows:

(a) Unit, intermediate, and shipping containers should be secure and free from leaks, rust or other contaminants, dents, bulges or other distortion.

(b) Liquid products should be homogeneous solutions with no separation, sediment, or other degradation of components; contents should not have evaporated.

(c) Solid materials (e.g., powders) should be free flowing with no significant water absorption or other contamination.

(d) Aerosols/sprays should spray as designed.

(e) Markings should be securely attached, clear, and legible.

(f) The material shall be capable of performing its intended function.

(4) Upon determining that the shelf-life may be extended, the person doing the inspection/test will update the item tags, labels and markings to reflect the inspection/test date, new expiration date and name of person conducting the test. The following provides an example of a shelf-life extension label that can be utilized for this purpose:

Manuf/Cure Date	_____
Inspect/Test Date	_____
New Exp Date	_____
Authority, Stamp or Signature	_____

g. When ordering shelf-life materials, ensure appropriate shelf-life data is inputted to the Industrial Logistics Support Management Information System (ILSMIS) electronic stub requisition.

h. Segregate, identify, and maintain on-hand no more than a 30-day supply, as practical, of shelf-life materials.

i. Review on-hand shelf-life materials for adequate and correct marking and identification. Prepare and affix appropriate shelf-life labels and/or extension labels in accordance with paragraphs 2.1.2d and/or 2.2f(4) to any material that is not identified, or when the label is not clearly legible.

j. Maintain a file of a completed MSDS for chemical shelf-life materials in storage or usage.

k. Maintain surveillance of shelf-life materials to ensure:

(1) On-hand stocks are not beyond the expiration date.

(2) Shelf-life materials are serviceable.

(3) The quantity of shelf-life materials is limited to that which is necessary to support a unit of production. A unit of production should be a definable segment, usually no greater than 30 working days in length.

l. Prepare, in accordance with applicable procedures, and forward to the appropriate action point a Discrepancy Record (DR) for the disposition of shelf-life materials received in an unsatisfactory condition, with an expired shelf-life or with an unacceptable amount of remaining shelf-life.

m. Contact Public Works personnel for assistance in handling or disposal of hazardous materials or questionable shelf-life materials. Provide proper containers and packaging materials and the expertise necessary to prepare hazardous materials for disposal/pick up.

n. Provide justification, as requested by the Supply Directorate, for maintaining pertinent shelf-life materials as active stock items in stores.

o. When appropriate, perform an evaluation to assess the need for recall of products discovered to have been produced with, or contain, materials whose shelf-life (or cured shelf-life) has expired or is expected to expire before the intended service life of the product.

q. Advise the Supply Directorate when any shelf-life material fails prior to the assigned expiration date.