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Quality Assurance Project Plan

for

Mustard Gas Burial Ground Resource Conservation and Recovery Act Phase III RCRA Facility Investigation

Volume II of II

Naval Surface Warfare Center Crane

Crane, Indiana

Southern Division Naval Facilities Engineering Command

Contract Number N62467-94-D-0888

Contract Task Orders 0131 and 0158

June 2001

LABORATORY SOP TABLE OF CONTENTS

SOUTHWEST LABORATORY OF OKLAHOMA SOPs

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SWL-GA-100	6.0	08/15/00		Laboratory Quality Assurance Manual
SWL-GA-101	2.1	05/15/00	RVSD	Preparation, Review, Revision and Control of Documents
SWL-GA-102	2.0	10/05/00	RVSD	Information Systems Quality Assurance Program
SWL-GA-104	6.0	06/02/00	RVSD	Personnel Training
SWL-GA-105	1.0	09/09/99		Laboratory Corrective Action
SWL-GA-106	2.1	05/22/00	RVSD	Archival and Retrieval of Sample Documentation
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SWL-RD-122	2.0	05/18/99		Error Estimates and Minimum Detection Activities
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SWL-RD-141	4.0	07/14/00	RVSD	Radiation Safety

Edgewood Chemical Biological Center SOPs

(NEW)

Application of Gas Chromatography/Mass Spectroscopy (GC/MS) for the Detection of Military Agents Mustard and Lewisite, Nerve Agent Breakdown Products DIMP and DMMP, and Mustard Breakdown Products 1,4-Dithiane and 1,4-Thioxane in Water and Soil.

Application of Gas Chromatography/Mass Spectroscopy (GC/MS) for the Detection of Thiodiglycol (TDG) and Alkyl Methylphosphonic Acids in Soil and Water Samples.

SOUTHWEST LABORATORY OF OKLAHOMA, INC. *and*
AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.

TM

Standard Operating Procedure

Preparation, Review, Revision, And Control of Documents

Document No.: SWL-GA-101

Rev No. / Date: Rev. 2.1 — 05/15/00

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APPROVALS	DATES
Procedure Prepared By	May 15, 2000 Date
Laboratory Manager or Director	05/18/2000 Date
Laboratory Safety Officer	5/16/2000 Date
Laboratory QA/QC Officer	May 16-2000 Date

(Effective Date is 10 calendar days after the last signature above - QA/QC Officer)

Document Status

[ ] Controlled DC No. _____ Issued to: _____ Date: _____ QA: _____

[✓] Official Copy OC No.: 2431 Issued to: Linda Lasonovich Date: 11/14/00 QA: _____

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PREPARATION, REVIEW, REVISION, AND CONTROL OF DOCUMENTS

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1.0 PURPOSE AND APPLICATION

1.1 The purpose of this document is to set forth guidelines for the preparation, review, approval and distribution control of Standard Operating Procedures (SOPs) affecting the quality and consistency of the work performed by Southwest Laboratory of Oklahoma, Inc.

1.2 Written documents shall be prepared and approved for all activities affecting the quality and consistency of work performed by Southwest Laboratory of Oklahoma. Activities will include, but are not limited to receipt, handling, preparation, and analysis of samples; use and maintenance of standards; calibration and performance checks of measurement equipment; reduction, evaluation, and reporting of data; the archival and retrieval of raw data, supporting documentation and electronic media generated during analysis; and general laboratory practices which support these activities. In addition, procedures shall be written, which conform to government regulations affecting worker health and safety, hazardous waste management, and radiation protection, and shall be governed by the same controls as those affecting quality.

2.0 RESPONSIBILITIES

2.1 The appropriate personnel shall be designated by the Laboratory Manager/Director or his/her designee to author each procedure.

2.2 Procedures, which are administrative or non-analytical in nature, fall under the category of "General" procedures and shall be reviewed and approved by the QA/QC Officer, Laboratory Manager/Director, and Laboratory Safety Officer. All other SOPs are categorized as "Technical" or analytical and shall be reviewed and approved by the responsible Laboratory Program Manager/Section Supervisor; Laboratory Safety Officer; and the QA/QC Officer.

2.3 All laboratory staff shall perform their tasks according to the most current written and approved procedures.

2.4 All laboratory procedures shall be reviewed annually. Procedures not requiring any changes will be so marked on the annual review form (Attachment 1). A copy of this form will be distributed documenting the review for all controlled copies of the SOP.

2.5 All revisions to existing procedures are subject to the same preparation, review, and approval system as new original procedures.

2.6 All procedures/documents requiring "Retired" status must be indicated on the "*Request for New, Revised, or Retired Documentation*", and shall be routed to the QA-DC Designee for processing and distribution of retired inserts.

2.7 The QA Department shall assign the responsibilities of Document Control to a member of its department's staff, who must be proficient in the use of MS Word, MS Excel, and Windows Explorer. The designated individual will be responsible for the controlled distribution, and maintenance of all documents as described herein.

3.0 EQUIPMENT

MS Office 97 software; Velo-Binder; Document Stamps

4.0 PRECAUTIONS

Controlled documents, which are not recovered: Reference, SWL-GA-105. *"Corrective Actions"* for instructions.

5.0 PROCEDURES

5.1 Initiation of New SOPs

The implementation of a new SOP is contingent on necessity to be determined by the QA/QC Officer, Program Manager/Section Supervisor, or Operations Manager, and is based on EPA or Client's specific requirements or in response to auditors findings.

5.1.1 When General procedures are required for an activity, the QA/QC Officer or Laboratory Manager/Director shall assign the responsibility for authoring the procedure to a staff member familiar with the required function.

When a technical procedure is required for an activity, the appropriate Program Manager/Section Supervisor is responsible for authoring the procedure.

5.1.2 In the case of both new G series general procedures and new O, I, or R series technical procedures, a "*Request for New, Revised or Retired Documentation*" (commonly referred to as "Revision Request Form" — see Attachment 1) must be filled out, indicating the justification for implementation of this procedure. This form is comprised of several sections: General SOP information, New SOP information, SOP Revision information, Retired SOP information, and a section for signatures at the bottom of the form.

Note: For New SOPs, the Document # portion of the form shall be filled out by the QA-DC Designee. Any suggestions for the Document #, by the Supervisor/Author shall be noted in the comments section.

This form will follow the draft document to the QA Department where the draft will be completed in the appropriate format (see Attachment 1).

5.1.3 The staff member assigned to creating the new SOP shall research necessary technical information to assure that an adequate procedure is written and then shall generate the initial draft supported by this research.

5.1.4 The designated author shall follow the outline for required text sections for general and technical procedures as referenced in Attachment 1.

5.1.5 The designated author shall include all necessary instructions, sample forms, and drawings necessary to properly complete the procedure. When applicable, the

author shall also specify or reference appropriate quantitative or qualitative acceptance criteria to ensure that prescribed activities are satisfactorily accomplished. This shall include applicable QC information as applies to the subject SOP. Acceptance criteria shall be the same as that for similar analytes or methods (spike recoveries, duplicate results, method detection limits, and chemical separation). Alternatively, acceptance criteria should be comparable to independently determined results (e.g. published in SW846, EPA 600, Standard Methods, or other credible sources.)

5.1.6 The draft document (and Revision Request Form) shall be submitted to the appropriate Program Manager/Section Supervisor for approval. Once approved, the draft document shall be submitted to the QA Department for assignment of an SOP number and formal publishing. The QA department will insure that all final formal documents follow the guidelines for consistent formatting of all procedures as stated in Attachment 1.

5.1.7 Once formally edited and printed by the QA-DC Designee, the electronic file shall be saved to a predetermined location in the QAQC directory and drive.

NOTE: In order to maintain control of all documents, only QAQC Department personnel shall be allowed access to the directory mentioned in the previous paragraph. If electronic files of drafts need to be submitted to the Designee, the file can be e-mailed.

The document will be circulated for review by and for approval signatures from the author (see *Request for New, Revised, and Retired Documents* form for identity), the Program Manager/Section Supervisor, QA/QC Officer, Laboratory Safety Officer, and/or Laboratory Manager/Director. At any time during circulation for signatures, the SOP may be returned to the QA-DC Designee for additional corrections/changes and then resubmitted for approval signatures. All circulating documents shall be covered by a brightly colored, boldly marked transmittal indicating "SOP ATTACHED", to reduce the risk of misplacement.

5.1.8 The "Approved" document is returned to the QA Department for distribution. (See Section 5.4)

NOTE: The SOP is not considered an official "Master" copy until it has been returned to the QA-DC Designee with all approval signatures in place.

5.2 Revision of Existing SOPs

Revisions of existing SOPs are to be performed as needed, and will be determined by Laboratory personnel, Program Manager/Section Supervisor, or QA/QC Officer, based on annual SOP review (see "Overdue Standard Operating Procedures" report, Attachment 1), identification of any errors, EPA or Client's specific requirements, or in response to auditors findings.

Note: Forms and logbooks are considered part of a SOP. Any change or implementation of forms or logbooks shall follow the procedures outlined in this document. See sub-sections 5.8 through 5.9 for specifics on logbook/forms control and maintenance.

5.2.1 The revision request form must be completed and submitted to the QA/QC Designee. If no changes are required (as in the case of SOPs up for Annual Review but requiring no changes), the form must be completed and specified that this was an "Annual Review" and that no changes were required. If changes are minor, use the space provided in the description portion of the form. If necessary, attach any additional instructions or related forms to this request.

5.2.2 The QA/QC Designee will process all requests in order of priority as listed on the "Overdue Standard Operating Procedures" report.

5.2.3 Once completed the Designee will save the electronic file to a predetermined location in the QA/QC directory and drive.

NOTE: In order to maintain control of all documents, only QAQC Department personnel shall be allowed access to the directory mentioned in the previous paragraph. If electronic files of drafts need to be submitted to the Designee, the file can be e-mailed.

The document will be circulated for review by and for approval signatures from the author (see *Request for New, Revised, and Retired Documents* form for identity), the Program Manager/Section Supervisor, QA/QC Officer, Laboratory Safety Officer, and/or Laboratory Manager/Director. At any time during circulation for signatures, the SOP may be returned to the QA-DC Designee for additional corrections/changes and then resubmitted for approval signatures. All circulating documents shall be covered by a brightly colored, boldly marked transmittal indicating "SOP ATTACHED" (see Attachment 1), to reduce the risk of misplacement.

5.2.4 The designee must use the "SOP Progress Sheet" (Attachment 1) to briefly describe the work performed, or to note any unusual occurrences; log the date procedure was finalized; and record the revision number.

The designee must also log the document number, name of person SOP was given to, and the date the SOP was submitted for signatures (see "Manual Follow-up of New Revisions Circulated for Signatures").

If the SOP is listed on the "Overdue SOP" report, the designee must mark out the SOP Document ID with an orange highlighter, to indicate that the QA/QC Designee has updated this SOP.

5.2.5 Once SOP is returned to the designee with all approval signatures in place, and no additional corrections are required, the SOP is then ready for distribution (see Section 5.4).

NOTE: The SOP is not considered an official "Master" copy until it has been returned to the QA-DC Designee with all approval signatures in place.

5.3 Retirement of Existing SOPs

An SOP must be retired if we no longer perform the procedure described therein; if the procedure is still performed, but mentioned in another SOP; and whenever the existing revision is replaced by a more current revision.

5.3.1 When the end-user (laboratory personnel), Supervisor, QA/QC Officer, or Operations Manager decide that a particular procedure is no longer valid or necessary, that person must complete the "Request for New, Revised, or Retired Documentation" form.

The Document Title, Document Number, etc. must be completed, as well as the lower portion labeled "Retired". The word "Procedure" must be circled.

5.3.1.1 The form is then submitted to the DC-Designee who will process this request.

5.3.1.2 The designee will generate a "Retired" cover insert using the template file "Retircvr.doc", and specifying that this is a retired procedure.

The "Retired" cover is kept with the revision request form until time for distribution (see Section 5.4).

5.3.2 If a procedure is still performed and is mentioned in more than one SOP, the supervisor may decide to retire one document and reference another. The "Request for New, Revised, or Retired Documentation" form shall be completed as instructed in section 5.3.1; however, the word "Document" must be circled and the SOP to be referenced must be noted in the space provided. Next, follow steps 5.3.1.1 and 5.3.1.2., making sure to choose "Document" and to reference the SOP that will remain, which also contains the same procedural information.

5.3.3 Procedures being replaced due to implementation of a newer revision shall follow retirement procedures as instructed in Section 5.4.5, but not before undergoing Section 5.2.

5.4 SOP Distribution Procedures

Once all newly revised SOPs are back in the QA/QC Department with all approval signatures in place, the QA-DC Designee will prepare them for distribution.

5.4.1 All original copies (copies which contain original signatures) are labeled "MASTER" at the bottom section of the cover page.

5.4.2 The Designee will gather together all Master SOPs in the same Series to be distributed in the same group.

5.4.3 The Designee shall generate an Acknowledgement Letter detailing the Document ID, Document Title, Revision Date, and Quantity of controlled copies to be distributed (as well as the Index information), for each revised series. An

Acknowledgement letter shall be generated for each specified recipient (see Master Distribution List in Attachment 1). Also generated is an updated series Index (Attachment 1), which lists all sops in a respective series, and highlights all the sops just recently revised. The Acknowledgment letter, series Index, and controlled SOP copies must be attached together for distribution.

5.4.3.1 The Acknowledgement Letter is a Microsoft® Word 97 document and is linked with a Microsoft® Excel 97 spreadsheet containing a listing of all the names of the recipients.

The Acknowledgement Letter must be updated and then merged with the Excel spreadsheet (which opens when the Word document is opened). Once the newly merged Word document is generated, specific distribution requirements for the recipients must be noted on individual letters (i.e. a recipient may require 2 or more controlled GA100 SOPs while everyone else receives only 1).

The electronic file containing the letters for each recipient is then saved to a predetermined location, and is then printed and signed in the designated portion of the letter.

5.4.3.2 Like the Acknowledgement Letter, the Series Index is a Word document linked with an Excel spreadsheet.

All new, newly revised, or retired SOPs are highlighted and then merged, saved, printed, initialed, and stamped.

5.4.4 The Master SOP is then used to make the total required number of copies for distribution (see Master Distribution List – SOP Series for specific quantity information, etc.). Each copy must be stamped with the "Controlled Copy" stamp; have the "Controlled" option in the "Document Status" section completed; and grouped with the corresponding Index and Acknowledgement Letter.

Once Sections 5.4.3.1 through 5.4.3.2 are completed for each Series, the SOPs are logged in the "Acknowledgement Letter and Index Distribution Log" and in the Microsoft® Outlook™ Calendar Task Pad (with recipients names listed) for follow-up of distribution responses.

The controlled copy packages are then distributed to the recipients. Each recipient is given 10 calendar days to return the retired revisions with the Acknowledgement letters and old indices. All old indices and cover pages with the "Controlled Copy" stamp are shredded while the Acknowledgement letters with the recipients signatures are filed. The recipient's name is taken off of the Calendar Task Pad.

NOTE: After 10 days, the Calendar Task Pad will remind me of all outstanding Acknowledgement letters. This reminder shall be forwarded to all individuals remaining on the list.

5.4.5 The old Master copies shall be pulled out of the Master Copy binders; stamped with the "Retired" stamp; dated; and filed in "Retired SOPs" drawer.

NOTE: Prior to filing, be sure that the original revision request forms remain in the Master Copy binders. The new Master SOP document will be placed in the Master Copy binder along with all the prior revision request forms.

5.4.6 After new revisions are implemented, the Excel spreadsheet titled "SOP List" shall be updated. All existing SOPs are listed in this spreadsheet, and any new SOPs are added. When a SOP has been revised and distributed the new date and revision number shall be updated in the SOP List. If the SOP is a new procedural document, the SOP Title, Revision Number, and Revision Date shall be added to the list. The revision dates on this list are good for one year, at which time the SOPs will be due for the Annual Review.

NOTE: This workbook consists of four spreadsheets: Active; Overdue; Two-month Outlook; and Cover.

5.5 Scheduled Review of Controlled Documents

5.5.1 The QA DC-Designee shall maintain the Excel workbook titled "SOP List" (see Section 5.4.6).

5.5.2 Each Monday morning, a spreadsheet report showing all SOPs overdue up to Monday's date, shall be generated from this file.

NOTE: All SOPs older than one year shall be considered "Overdue" for its Annual Review.

Also generated from this file shall be the Two-Month Outlook. These two reports combined with the QC Tracking Cover Sheet shall be submitted to the Operations Manager.

NOTE: See Operations Manager for quantity of reports to submit.

5.5.3 These reports are distributed to the supervisors attending the weekly staff meeting, who will locate, review and update all SOPs on the list pertaining to them.

They shall follow the procedures outlined in Section 5.2 of this SOP for Revision of Existing SOPs.

5.5.4 The SOPs will remain on this list until they are updated and given a new revision date.

5.5.5 This process is done on a weekly basis throughout the year.

5.6 Logbook Maintenance and Control

5.6.1 Each logbook shall be maintained and controlled by the Program Manager or Section Supervisor to which its corresponding SOP applies. The QA department

shall maintain all logbooks which correspond to any G series documents. Both shall be maintained as described below.

5.6.2 Each Program Manager or Section Supervisor shall maintain a master log of each individual logbook that is currently being used in the respective departments and shall also use this master logbook to track department archival (filled or retired logbooks), and the ultimate transfer of custody of said logbooks to the QA department for archival/long term storage. When a department logbook is created for use, its Book ID number, SOP ID name, revision and date of logbook issuance shall be recorded in the " Department Master Logbook Log" as shown in Attachment 2. When a logbook is filled (or retired), the following columns shall be updated in the logbook; Date Completed (filled, retired, out of date, etc.), Department Storage Location (example: "OV Department Logbook Archive Storage File"), Date of transfer from department to the QA department for archival/long term storage; and, initials of the responsible Manager/Supervisor or his/her designee.

5.6.3 Each Program Manager/Section Supervisor's "Department Master Logbook Logs" shall be recorded in the "Laboratory Master Logbook Log" (see Attachment 2) by the QA department as part of its document control procedures.

5.6.4 The QA department shall maintain a traceable system to ensure proper archival and retrieval of all department logbooks as part of its "Archival and Retrieval of Sample Documentation" procedure (see SWL-GA-106 for details).

5.6.5 The Program Manager/Section Supervisor (or his/her designee) shall be responsible for the review of all logbook entries and shall initial and date the bottom of each log page upon his/her review. The designated reviewer shall review each logbook depending upon frequency of use (i.e.; daily, weekly, monthly, quarterly, etc.). As an option, each logbook shall include a review page (last page of the book) verifying the review of each page (page ranges may be entered), the reviewer's signature, and the date. In the case of either option, the Program Manager/Section Supervisor shall verify that the review of all pages are accounted for prior to transfer to the QA department for archiving.

5.6.6 See Attachment 3 for formatting instructions of Logbooks.

5.7 Forms Control

5.7.1 All forms are controlled by this SOP to maintain that the current authorized forms are only used at any given time in the laboratory.

5.7.2 All used forms (filled out as per it's corresponding SOP) become an important part of the data package information generated as they are used. These "used" forms shall be kept with appropriate data and maintained by each department just as raw data, and other documents pertinent to sample analyses are kept stored and archived (see SWL-GA-106 *"Archival and Retrieval of Sample Documentation"*).

5.7.3 All Forms or Logs, etc. must have the SWLO Logo, Company Name and Address, Title of Form or Log, etc. (See Attachment 3 for examples.)

6.0 RECORDS

6.1 The QA-DC Designee shall maintain the following records and provide the QA/QC Officer with a monthly reconciliation report for QA/QC of its document control procedures and systems.

6.1.1 Master index of all controlled procedures, with active revision number and issue date for each procedure.

6.1.2 Request for New or Revised Procedure Forms - All approved request forms shall be maintained with the Master copy of all active procedures.

6.1.3 Distribution lists which include all controlled copies

6.1.4 Receipt acknowledgment forms

6.1.5 Master copy of all active procedures

6.1.6 At least one copy of all retired revisions of procedures.

6.1.7 Master Logbook for "Scheduled Review of Procedures"

6.1.8 Master record of all "Department Master Logbook Logs"

6.1.9 Monthly Reconciliation Reports (approved and initialed by the QA/QC Officer or his/her designee).

7.0 REFERENCES

7.1 Naval Facilities Engineering Service Center, Interim Guidance Document, *"Navy Installation Restoration Laboratory Quality Assurance Guide, February 1996"*

7.2 Office of Nuclear Safety Policy and Standards, DOE Order 5700.6C: *"Quality Assurance Program, A Management System, October 1992, Revision 1"*

8.0 DEFINITIONS

8.1 Terminology

Controlled Document..... Those documents which are controlled by a system which limits access to creation and revision of said document. Limits distribution of documents by assigning individual responsibility of each document including signed receipt of current updates and destruction of replaced copies. Traceability of each document on the distribution list to

individuals responsible for said documents and universal distribution of any and all updates to all participants. Examples of "controlled" documents are SOP's and QA Manual.

Official Documents ..... Those documents that are officially authorized by management or other authority to be written, copied and distributed. Official documents may be assigned serial numbers and recorded as to distribution. These documents are not always (as controlled documents are) updated and distributed as the original is changed. Most Official copies are indicated as originals. Examples: copies of the QA Manual, SOP's, company letters on company letterhead, and memos.

Unofficial Documents ..... Those documents that are unofficially written, copied and distributed generally as educational materials. Examples: Faxes, draft or working copies of QA Manual, SOP's, Forms/logbooks, and any form developed unofficially to assist in day to day activities individually.

Nomenclature ..... Identification via a combination of: key words, classifications, labels, or designations (or specificity).

Ancillary ..... Additional or secondary to the primary, i.e.; ancillary equipment.

8.2 Acronyms

EPA ..... The United States Environmental Protection Agency

GC ..... Gas Chromatography

GC/MS ..... Gas Chromatography/Mass Spectrometry

HPLC ..... High Performance Liquid Chromatography

LCS ..... Laboratory Control Spike

MSD ..... Matrix Spike Duplicate

PE ..... Performance Evaluation Sample

PEST/PCB ..... Pesticides and PCB (Polychlorinated-biphenyls)

QA ..... Quality Assurance

QA/QC ..... Quality Assurance and Quality Control

SOP ..... Standard Operating Procedure

SWL ..... Southwest Laboratory of Oklahoma, Inc.

SW846..... Test Methods for Evaluating Solid Waste

9.0 ATTACHMENTS

9.1 Attachment 1 — Processing of SOPs

9.2 Attachment 2 — Logbook Forms

9.3 Attachment 3 — Formatting of Logbooks and Forms

ATTACHMENT 1
Processing of SOPs

Request for New, Revised, or Retired Documentation
Formatting of Standard Operating Procedures
Overdue Standard Operating Procedures Spreadsheet
"SOP ATTACHED" Cover
SOP Progress Status Sheet
Manual Follow-up of New/Revised SOPs Circulated for Signatures
Acknowledgement Letter
Procedures Index
Master Distribution List
Cover Page

Request for New, Revised, or Retired Documentation

GENERAL ADMINISTRATIVE

SOP SWL-GA-101, REV. # 2.0 FILE ID: GA101F1.doc

Document Title: _____

Document No.: SWL-___.___-___ ___ ___ ***Existing*** Rev. No. & Date ___.___—___ ___/___ ___/___ ___

Submitted By *(if other than supervisor)*: _____ Date Submitted: _____

Approved by: _____ Date: _____
Program Manager/Section Supervisor

NEW Procedural Document *(Please attach hard-copy, and/or note below if file was e-mailed.)*

Author: _____

Justification for New Procedure:

_____

_____

_____

REVISION *To Existing Procedural Documents, Including Forms & Logs*

Reason for Revision: ___**Annual Review** (___Changes / ___No Changes) ___**Other** (error found, etc.)

For Minor Revisions, provide the following References:

Page ____ Section: ____.____; Subpart: ____ Page ____ Section: ____.____; Subpart: ____

Page ____ Section: ____.____; Subpart: ____ Page ____ Section: ____.____; Subpart: ____

Attachment No. _____ — Title: _____

Attachment No. _____ — Title: _____

Required — The following must be completed if revisions are necessary.

Description:

_____

_____

_____

_____

RETIRED *(Circle One — **PROCEDURE** or **DOCUMENT**)*

Circle "Procedure" if we no longer do the Method/Procedure discussed in the SOP.
Circle "Document" if the Method/Procedure is now included in another SOP. (Reference Other SOP's below)

_____

_____

SOUTHWEST LABORATORY OF OKLAHOMA, INC.
1700 West Albany • Broken Arrow, OK 74012 • Office (918) 251-2858 • Fax (918) 251-2599

NOTE: Actual size is 8 ½ x 11 inches. (Standard letter size).

A1-1 Formatting of Standard Operating Procedures

A1-1-1 Nomenclature of SOP documents shall be as follows:

A1-1-1-1 All Documents shall be categorized within the following series. Its reference nomenclature shall include the laboratory acronym, section series and sequential numbers assigned (within each series) to each document. Following are the subject Series, descriptions, and examples of document ID number.

G General Series - Non-analytical and Administrative
"GA" General and Administrative SWL-GA-100
"GP" General Projects SWL-GP-100

O Organic Series - Extractions, GC, GC/MS, Dioxin, Air
"OG" GC SWL-OG-100
"OE" Organic Extractions SWL-OE-100
"OP" GC Pest/PCB SWL-OP-100
"OS" GC/MS Semivolatiles SWL-OS-100
"OV" GC/MS Volatiles SWL-OV-100
"OL" HPLC SWL-OL-100
"OA" GC/MS Air SWL-OA-100
"OD" GC/MS Dioxin SWL-OD-100

I Inorganic Series - Sample Prep, Metals, General Chemistry
"IN" Inorganics & Wet Chem SWL-IN-100

R Radiochemistry Series - Prep, Isotopes
"RD" Radiochemistry SWL-RD-100

A1-1-1-2 The index of each series (i.e.; GA Series Index, etc.) shall also be a "controlled" document. The index includes the document number, revision number, document title, and latest revision date and shall be retained in front of each controlled SOP binder for each series.

A1-1-2 Header Information: Every page of each procedure shall contain the following information in a header:

A1-1-2-1 The Laboratory's abbreviation: SWLO, INC./AATS, INC.

A1-1-2-2 Document Number (i.e.; SWL-GA-101)

A1-1-2-3 Revision Number and issue date (1ST digit major rev.: 2ND minor rev., i.e. Rev.: 2.1 — 12/12/98)

A1-1-2-4 Document Title

A1-1-3 Footer Information: Page Number and the total number of pages in the procedure shall be included in the "footer " (right justified), as well as the initials of the typist processing the revision (left justified). For Attachments, figures, tables, etc. the header information may be altered as necessary to avoid obscuring the information presented.

A1-1-4 Cover Page Contents: Each procedure's cover page shall contain the laboratory name, the formal document Title [including Method(s)], document ID Number, Revision Number and Revision Date. In addition, a signed and dated record of review and approvals with all relevant document control information must be present on the cover page. See page A-1-17 for an example of the required cover page.

A1-1-5 Required Text Sections for General Administrative Procedures: Each General procedure shall contain at a **minimum** the following sections:

1.0 Purpose and Application
(Intent of the procedure and its applicable activities)

2.0 Responsibilities
(Specific to this procedure, not general responsibilities)

3.0 Equipment
(Specialized equipment required for the specific procedure)

4.0 Precautions
(Warning of necessary safety and waste considerations, alerts for interferences, or decision points)

5.0 Procedure
(Step by step instructions, flow charts, decision criteria, as needed)

6.0 Records
(Documents required at completion of procedure, disposition, and quality control aspects)

7.0 References
(A list of documents which were consulted in the development of the SOP or which may provide further information on a particular subject.)

8.0 Definitions
(A glossary shall be included which defines terminology, acronyms, etc.)

9.0 Attachments
(Additional documents which are necessary to execute the procedure, i.e.; forms, logbooks, diagrams, flow charts, etc.)

OPTIONAL Sections:

Additional Sections which may be required shall be added to the end of the minimum required Sections listed above. Example of an Optional Section for General Procedures is Section 10.0 - Health & Safety.

A1-1-6 Required Text Sections for Technical Procedures: Each Technical procedure shall contain at a minimum the following sections:

1.0 Scope and Application
(Description of the sample collection, analytical or other technical activities to which it applies, and the matrix, chemical compounds of interest, and concentration levels if available shall be described. All methods the procedure is based on shall be mentioned here along with any method deviations (if applicable). In instances where multiple methods are used and the strictest QC is used, there must be a mention of this here as well.

2.0 Method Summary
(A brief description of the method.)

3.0 Health and Safety
(A description of any hazards to the health and safety of the user with specific references to the laboratory's Safety SOP and Hazardous Waste Management SOP.)

4.0 Sample Preservation, Containers, Handling & Storage
(A description of the amount of sample to be collected, the container type, chemical preservation (if required) and storage requirements)

5.0 Interferences and Potential Problems
(A description of environmental conditions, or non-targeted chemical that can cause interferences which the user should be aware.)

6.0 Equipment/Apparatus
(The names and model numbers of equipment and/or instruments used in the procedure, including ancillary equipment such as power supplies, data acquisition equipment and software, recorders, etc.)

7.0 Reagents
(List of all chemical reagents used in the procedure (i.e.; cylinder gases, acids, solvents, standards, powdered reagents), and a description of preparation methods of any stock solution (i.e.; 10% nitric acid, etc.).)

8.0 Procedure
(A step by step list of how the actual sample collection, analytical procedure or technical activity is performed.)

9.0 Calculations
(Equations/formulas for calculating results and associated units of measurement. If direct read-out this should also be stated.)

10.0 Quality Assurance / Quality Control
(List and frequency of QA/QC samples to be collected and analyzed with the environmental samples (i.e.; blanks, duplicates, spikes, and PE's). Acceptance criteria for each QA/QC sample should be given along with other QA/QC activities such as equipment calibration, background sample collection, and implementation of manufacturer's instructions. Include actions taken when a criterion is not met.)

11.0 Waste Disposal / Pollution Prevention
(If applicable, reference to the laboratory's Hazardous Waste Management SOP should be included, along with any additional information not included in the general Hazardous Waste SOP.)

12.0 Method Performance
(This section shall include the performance limits for LCS and MSD combinations as appropriate. Also Laboratory detection limits for most of the above shall also be included. Although the data included in the document will be fairly current, reference shall be made to the location of the computer directory for the <u>most</u> recent data available.)

13.0 References
(A list of documents which were consulted in the development of the SOP or which may provide further information on a particular subject.)

14.0 Definitions
(A glossary shall be included which defines terminology, acronyms, etc.)

15.0 Attachments

Attachments - Additional documents which are neeessary to execute the procedure, i.e.; forms, logbooks, diagrams, flow charts, etc.

OPTIONAL Sections:
Unique Method Information - Laboratory specific information, such as applicable LIMS test codes or any variances from published method references.

Header/Footer Information and Style

Southwest Laboratory of Oklahoma, Inc. GA-Series: SWL-GA-101	
Title: Preparation, Review, Revision, and Control of Documents	**Rev. No.: 1.0 — 05/24/97**

❶ ❷

❸ ❹

HEADER INFORMATION (EACH PAGE)

FIRST LINE

❶ Laboratory Name (Left Justified) ❷ Series and Procedure No. (Right Justified)

SECOND LINE

❸ Title of Procedure (Left Justified) ❹ Revision No/Date. (Right Justified)

Note: While clicked on the last line of the Header (Title: —) select from the "Borders" toolbar the thin double line and then select the bottom border.

❺

Typist: LLR	**Page 14 of 26**

FOOTER INFORMATION (EACH PAGE)

❻ Page No. of Total (Right Justified) [type in **Page** *{insert page # option from Header/Footer speed buttons}* then type **"of"** *{insert total # pages}*, i.e.: **Page 14 of 26**]

STYLES FOR BOTH HEADER AND FOOTER

FONTS	Times New Roman Size: 12 pt Header Bold	Size: 10 pt Footer
PARAGRAPH	Line Spacing: Select - Exactly: 12 pt	
CHARACTERS	Upper and Lower Case	

Note: The style is consistent with the exception of the point size between the Header (at 12 pt) and Footer (at 10 pt) due to page length considerations.

PROCEDURAL DOCUMENT FORMATTING SPECIFICATIONS

Fonts

Body of Text:	Times New Roman	Point Size: 12 pt	Style: Regular
Major Sections:	**Times New Roman**	**Point Size: 12 pt**	**Style: Bold**
Outline Numbers:	**Times New Roman**	**Point Size: 12 pt**	**Style: Bold**
Reference Titles:	*Times New Roman*	*Point Size: 12 pt*	*Style: Italicized*
Insert Notations:	Arial:	Point Size: 10 pt	Style: Regular

Margins

Top: 0.5"	Left:	0.75"	Header (see Attachment 2)
Bottom: 0.5"	Right:	0.75"	Footer (see Attachment 2)
Gutter: 0"			

Paragraphs

Line Spacing (all text):		At Least	12 pt
Paragraph Spacing:	Before	Section Headers:	18 pt
	Before	All other Subsections:	10 pt

Major Sections Lines: 6pt above Bold, All Caps 12pt font size

Title of Document 12 pt - Regular All Caps/Centered

PREPARATION, REVIEW, REVISION, and CONTROL OF DOCUMENTATION

TABLE OF CONTENTS

All Caps size 16pt Bold and Centered

1.0 PURPOSE AND APPLICATION 1
- **1.1** Purpose of Document 1
- **1.2** Application 1

2.0 RESPONSIBILITIES 1
- **2.1** Responsibility to Assign Author 1
- **2.2** Review and Approval Authority 1
- **2.3** QA Manual Review and Approval Authority 1

Leader tab set for dotted line.

Subsection line spacing at single. Bolded No.'s

Pagination for TOC is i, ii, iii.

i

Notes: ① **Please do not line space using the "Enter" (or Return key). Follow the guidelines setup above.**
② **When text wraps to next page make sure there is 12 pt line spacing between the Header and text body. This ensures readability and avoids a "crammed" appearance.**
③ **When typing a formal name or title for a reference book, logbook and forms, etc. quotation marks should be used, i.e.;** *"Request for New or Revised Procedure Form"*.
③ **Including subsections on the Table of Contents is not necessary when the SOP has few pages.**

PROCEDURAL DOCUMENT FORMATTING SPECIFICATIONS

EXAMPLE

Major Section Headers Flush

Paragraph at 18 pt

Paragraph at 10 pt space

Paragraph at 10 pt space

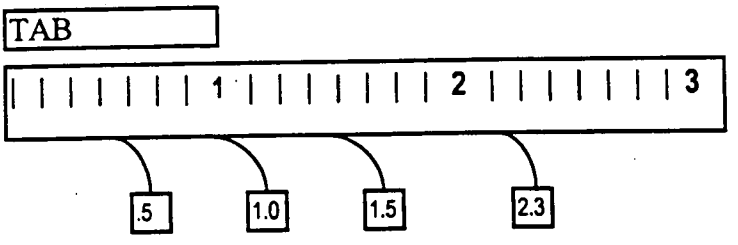
5.0 PROCEDURES

5.1 Format of Standard Operating Procedures

5.1.1 Nomenclature of SOP documents shall be as follows:

5.1.1.1 All Documents shall be categorized within the following series. Its reference nomenclature shall include the laboratory acronym, section series and sequential numbers assigned (within each series) to each document.

Keep the numbering system to 4 levels only. A rule of thumb is – "if you don't have a B you shouldn't have an A". In SWL's numbering format this would mean: if you don't have a 5.1.2 you shouldn't have a 5.1.1 – if that was the case one would combine the information in 5.1 or drop down under 5.1 with small cap (a), (b), etc.. In the example above the (a) would align with the 1.0" tab setting and indent only 2 cm's. A bulleted list is also acceptable.



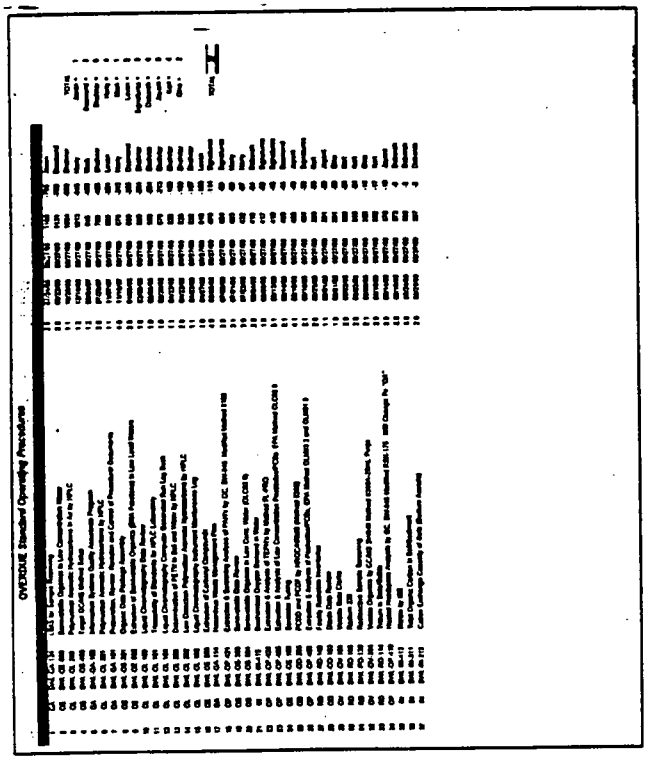
1st Level **1.0** → → →**0.5"** - SECTION TITLE Hanging Indents will be same as start of text

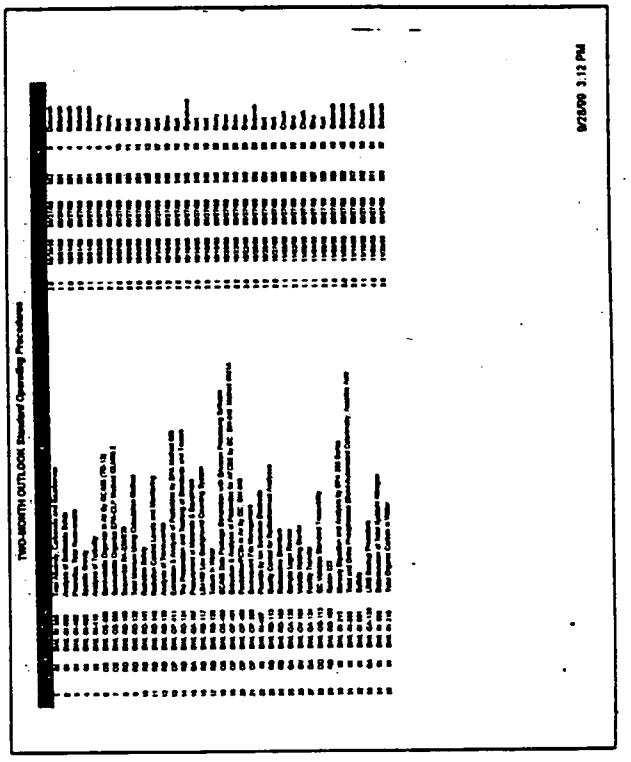
2nd Level No **1.1**→ →**0.5"** → →**1.0"** - Text - Hanging Indent at 1.0"

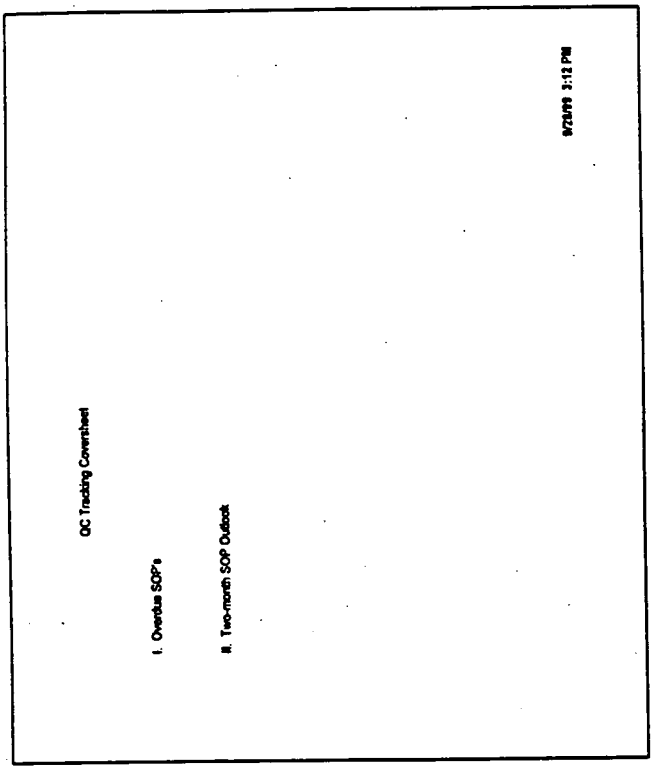
3rd Level No. **1.1.1**→ → → → **1.0"** → **1.5"** - Text - Hanging Indent at 1.5"

4th Level No. **1.1.1.1**→ → → → **1.5"** → **2.3"** - Text - Hanging Indent at 2.3

Note: actual measurement is between the first and second hash mark after the 2.3







Overdue Standard Operating Procedures Report:

- QC Tracking Cover Sheet
- Overdue SOPs
- Two-month Outlook

NOTE: Actual size of report is 8½ x 11 inches (standard letter size).

Original is on Bright Pink Paper

SOP ATTACHED

In order for the SOP Department to maintain accurate records of which SOPs are currently being reviewed for approval, and at what stage of approval they are in, we are asking everyone whose signatures are required for this SOP to notify Levan LaRue by phone (at ext. 120) that you have signed it and to whom you forwarded the SOP for additional signatures.

Document # :_____
Time :_____
Date :_____
Initials :_____

NOTE: Actual size is 8 ½ x 11 inches. (Standard letter size).

Typist: LLR

Original is on Light Yellow Paper

SOP Progress Status Sheet

DATE/TIME	STATUS

NOTE: Actual size is 8 ½ x 11 inches. (Standard letter size).

Typist: LLR

Manual Follow-Up of New/Revised SOPs Circulated for Signatures

Doc. No.	Passed on To:	Date	Passed on To:	Date	Passed on To:	Date	Passed on To:	Date	Given to Document Control / Date

Soptrack

NOTE: Actual size is 8 ½ x 11 inches. (Standard letter size).

Typist: LLR

Receipt of Controlled Document Acknowledgment Letter

SOP SWL-GA-101 Rev. 2.0 File ID: G-ackmrg.doc (For Ga-Series)

September 29, 1999

Name: Chuck Hoover
Title : QA/QC Officer
Dept : QA Department

Dear Mr. Hoover:

Enclosed are document controlled copies of the following new or revised procedures. Please read the following acknowledgement and verify that all documents listed are attached.

ACKNOWLEDGMENT

I hereby verify that I have received the procedural document(s) listed below, under my assigned Document Control Number DC001. I understand that I must acknowledge receipt within 10 calendar days of the date of this letter.

Please sign below, attach your retired version (if applicable) and return to the undersigned.

Procedure ID	Title	Rev No.	Issue Date	Copies
SWL-GA-105	Laboratory Corrective Action	1.0	09/09/99	1
SWL-GA-111	Laboratory Safety Plan	3.3	09/13/99	1
SWL-GA-116	Bottle Preparations	3.2	09/22/99	1
SWL-GA-139	Maintenance and Management of the LIMS System	1.0	09/15/99	1
SWL-GA Series	INDEX (revised as of 9/29/99)	N/A	9/29/99	1

Signature

Date

Sincerely,
Southwest Laboratory of Oklahoma, Inc.

Levan LaRue,
QA Document Control Designee

09/29/99 at 9:09 AM

NOTE: Actual size is 8 ½ x 11 inches. (Standard letter size).

General and Administrative Procedures Index, SWL-GA-101

SOP SWL-GA-101 Rev. # 2.0 File ID: 04DXMSTR.doc (For GA-Series)

G-SERIES INDEX

Document Control No.: «DC» Name: «Name» QA Initials: ____

Old ID	New ID	Rev	Document Title	Issued
SWL-GA-100	SWL-GA-100	3.6	Laboratory Quality Assurance Manual	07/29/99
SWL-GA-101	SWL-GA-101	1.1	Preparation, Review, Revision, Control of Procedural Documents	11/07/97
SWL-GA-102	SWL-GA-102	1.2	Information Systems Quality Assurance Program	06/04/97
[redacted]				
SWL-GA-104	SWL-GA-104	4.0	Personnel Training	02/24/99
SWL-GA-105	SWL-GA-105	1.0	Laboratory Corrective Action	09/09/99
SWL-GA-NEW	SWL-GA-106	1.1	Archival and Removal of Sample Documentation	01/13/99
SWL-GA-107	SWL-GA-107	1.2	Procurement of Materials & Equipment	10/16/98
SWL-GA-108	SWL-GA-108	4.1	Performance & System Audits	07/27/99
SWL-GA-109	SWL-GA-109	1.2	Traceability of Standards	09/15/99
SWL-GA-110	SWL-GA-110	3.2	Laboratory Documentation of Sample Custody	07/27/99
SWL-GENSAFT	SWL-GA-111	3.3	Laboratory Safety Plan	09/13/99
SWL-GA-112	SWL-GA-112	2.2	Control Charts	04/20/99
SWL-GA-113	SWL-GA-113	3.3	Definition and Determination of Detection Limits	09/12/99
SWL-GENHAZW	SWL-GA-114	4.0	Hazardous Waste Management Plan	06/05/99
SWL-GENCUST	SWL-GA-115	5.2	Sample Custodian	07/28/99
SWL-GA-116	SWL-GA-116	3.2	Bottle Preparations	09/22/99
[redacted]				
SWL-GENBALN	SWL-GA-120	4.1	Calibration of Electronic Balances	12/22/99
SWL-GA-121	SWL-GA-121	3.2	Glassware and Labware Cleaning	05/10/99
[redacted]				

Last Revised: 09/29/99 at 9:07 AM Page 1 of 2

General and Administrative Procedures Index, SWL-GA-101

SOP SWL-GA-101 Rev. # 2.0 File ID: 04DXMSTR.doc (For GA-Series)

G-SERIES INDEX (continued)

Document Control No.: «DC» Name: «Name» QA Initials: ____

Old ID	New ID	Rev	Document Title	Issued
SWL-HAZPLAN	SWL-GA-123	3.1	Hazardous Materials Contingency Plan/ Communication Plan	07/30/99
SWL-GENLIMS	SWL-GA-124	2.0	LIMS for Sample Receiving	07/24/99
SWL-GENWATR	SWL-GA-125	2.1	Laboratory Water Supply	06/03/99
[redacted]				
SWL-GENPREV	SWL-GA-127	3.1	Preventive Maintenance	09/09/99
SWL-GA-128	SWL-GA-128	3.1	Maintenance of "ZONE" Log Books	09/09/99
[redacted]				
SWL-GENRESP	SWL-GA-130	2.1	Respirator Protection Plan	06/30/99
SWL-GA-NEW	SWL-GA-131	2.0	Project Management	05/24/99
SWL-GA-NEW	SWL-GA-132	4.0	Data Reporting: Non-EPA	09/11/99
SWL-GA-133	SWL-GA-133	2.1	Usage of Blanks and Backup Blanks	09/15/99
SWL-GA-NEW	SWL-GA-134	1.1	Facility Transfer	11/02/99
SWL-GA-NEW	SWL-GA-135	1.1	Sample Login Review	11/02/99
SWL-GA-NEW	SWL-GA-136	3.1	Sending Data Packages to Agencies	03/24/99
[redacted]				
SWL-GA-NEW	SWL-GA-138	1.1	LIMS Backup Procedures	11/19/99
SWL-GA-NEW	SWL-GA-139	1.0	Maintenance and Management of the LIMS System	09/15/99
SWL-GA-NEW	SWL-GA-140	1.1	Homogenization of Soil, Sediment, and Sludge Samples	01/12/99
SWL-GA-NEW	SWL-GA-141	1.0	Testing of Extractable Organic Spike Solutions	03/24/99

DC No.: «DC» Issued to: «Position» Name: «Name» DC Designee: L. LaRue

Arrow plus Bolding indicates revisions to index

Last Revised: 09/29/99 at 9:07 AM Page 2 of 2

NOTE: Actual size is 8 ½ x 11 inches. (Standard letter size).

Typist: LLR

MASTER DISTRIBUTION LIST - SOP SERIES

Standard Distribution Information for SWL Controlled Documents.

GA (General & Administrative) Procedural Documents

ADMINISTRATIVE NAME	DC NO.	TECHNICAL NAME	DC NO.
DC Dept. receives 1 (non-controlled blank copy)		Harry Borg	(DC-004)
Chuck Hoover	(DC-001)		
Rob Harris	(DC-003)	Drew Cowan	(DC-005)
Mike Butler RETIRED	(DC-010)	Gina Jackson	(DC-006)
Steve Markham	(DC-012)	J. Shringarpure	(DC-007)
Jan Travis	(DC-014)	Deborah Inman	(DC-008)
Mark Smith	(DC-015)	DC009 receives all GAs incl. 4-controlled copies of GA100	
Keith Sims or Designee	(DC-017)		
Sarah Hodson (Sample Receiving)	(DC-023)	Kart Barface (DC-009) (DC-021, DC-022 for RD)	
DC-023 Receives 3-Controlled Copy of:		DC-009 also receives "1-extra Controlled Copy" of:	
SWL-GA-100 SWL-GA-109 SWL-GA-110 SWL-GA-111		6-Series incl. GA100, GA110, GA114, & GA119	
SWL-GA-114 SWL-GA-115 SWL-GA-116 SWL-GA-123		Shahriar Shahreza	(DC-011)
SWL-GA-124 SWL-GA-127 SWL-GA-129 SWL-GA-130			
SWL-GA-134 SWL-GA-135		Desmond Foster	(DC-016)
		DC016 receives All GA's, plus 3-GA121	
Randy Staggs	(DC-024)		
Beth Dass Retired	(DC-025)	Terry Martinelsis Retired	(DC-019)
		K.M. Bagaveedoss, Ph.D.	(DC-020)
Sandi Grovenstein	(DC-026)		
DC-026, DC-025, & DC-020 Each receive 1-Controlled Copy of the following:			
SWL-GA-109 SWL-GA-116 SWL-GA-121 SWL-GA-135			
Barbara Forrester	(DC-027)		
Pauline Lewis	(DC-028)		
Receives GA100			

%SWLDRGADCNAYYS\DCDA\RECORDS\DISTRIB\MSOPDISTR.DOC 09/29/99 at 9:30 AM

IN (Inorganics) Procedural Documents

ADMINISTRATIVE NAME	DC NO.	TECHNICAL NAME	DC NO.
Chuck Hoover	(DC-001)	Desmond Foster	(DC-016)
Steve Markham	(DC-012)	(DC-016 receives IN-499 & IN-504)	
Deborah Inman	(DC-008)		
Barbara Forrester	(DC-027)		

IN Series - Number of Copies

DC008 maintains TWO (2) Master Books on ALL Inorganic SOP's
See attached Reference List for the ADDITIONAL bench copies required (these are in ADDITION to the 2 sets listed above.

RD (Radiochemistry) Procedural Documents

ADMINISTRATIVE NAME	DC NO.	TECHNICAL NAME	DC NO.
Chuck Hoover	(DC-001)	Kart Barface	(DC-009, DC-021, & DC-022)
Steve Markham	(DC-012)		
Barbara Forrester	(DC-027)		

RD Series - Number of Copies

Kart gets just 1-copy for each DC9
Volatilized DC022

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O-Series (Organic) Procedural Documents

ADMINISTRATIVE NAME	DC NO.	TECHNICAL NAME	DC NO.
Receives ALL O-Series Documents			
Chuck Hoover	(DC-001)	Harry Borg	(DC-004)
Steve Markham	(DC-012)		
Barbara Forrester	(DC-027)		

Receives Individual O-Series Subparts (as indicated)

OA (Air) — Jayant Shringarpure (DC-007) — 3-Sets

[redacted]

OE (Organic Extractions) — Desmond Foster (DC-016) — 2-Sets

[redacted]

OL (HPLC) — Shahriar Shahreza (DC-011); Desmond Foster (DC-016) — 2-Sets OL200 — OL204, & OL206

[redacted]

OS (Semivolatiles) — Harry Borg (see above); Desmond Foster (DC-016) — 2-Sets (OS201 & OS206)

[redacted]

O-Series - Number of Copies

All O-Series DC Number holders generally require only one copy. Exception - C. Hoover (2)
Check with Individual Section Supervisors.

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(Standard letter size).

ATTACHMENT 1

IN-SERIES
DOCUMENT CONTROLLED COPIES

Example

2 Master Sets

PLUS

5 additional copies of SWL-IN-100

TOTAL: 6

No.	Doc. I.D.	Document Title
4	SWL-IN-100	Calibration of Equipments
2	SWL-IN-102	Inorganic Data Package Assembly
9	SWL-IN-103	Inorganic Preventive Maintenance Schedule
3	SWL-IN-104	Inorganic Traceability of Standards
2	SWL-IN-105	Digestion/Distillation Log
2	SWL-IN-106	1st & 2nd Data Review of ICP, GFAA, Mercury & Cyanide
3	SWL-IN-107	1st & 2nd Data Review for Wetlab Analyses
3	SWL-IN-200	ICP ILM04.0 Digestion and Analysis
2	SWL-IN-201	GFAA ILM04.0 Analysis and Digestion
2	SWL-IN-202	Mercury ILM04.0 Digestion and Analyses
4	SWL-IN-205	ICP SW3010A / 3050B Digestion and SW6010B Analyses
2	SWL-IN-206	Digestion of Aqueous/Soil Samples, GFAA, SW846-3020A/3050A
2	SWL-IN-207	Mercury, SW846-7470A / 7471A, Digestion and Analyses
2	SWL-IN-210	GFAA, SW846-7000 Series Analysis
2	SWL-IN-212	Cation Exchange Capacity of Soils (Sodium Acetate)
1	SWL-IN-215	Mercury Digestion and Analyses by EPA 200 Series
2	SWL-IN-218	Acid Digestion for Air Filters for Metals Analyses
2	SWL-IN-301	Anions in Water by IC by DIONEX 2010i
2	SWL-IN-302	Cyanide Distillation and Analysis for SW846-9012A
2	SWL-IN-303	Cyanide Distillation and Analysis for ILM04.0
1	SWL-IN-304	Nitrate-Nitrite/Nitrite (Colorimetric, Automated, Cadmium Reduction)
1	SWL-IN-305	Total and Ortho Phosphorous (Semi-Automated Colorimetry, Ascorbic acid)
1	SWL-IN-306	Phenolics in Water (Colorimetric, Automated, 4-AAP / Distillation)
2	SWL-IN-307	Analyses of Cyanide (SPECTROPHOTOMETER)
2	SWL-IN-308	Cyanide Amenable to Chlorination (IN122)
2	SWL-IN-309	Reactivity Cyanide (IN125, HW905)
1	SWL-IN-310	Total Organic Carbon in Water
1	SWL-IN-311	Total Organic Carbon in Soil/Sediment
1	SWL-IN-312	Determination of Total Organic Halides (TOX)
2	SWL-IN-314	Reactivity Sulfide (IN305, HW905)
1	SWL-IN-400	Ammonia Nitrogen by ISE
1	SWL-IN-401	Specific Conductance
1	SWL-IN-402	Phenolics, Total Recoverable
1	SWL-IN-403	Chemical Oxygen Demand Analysis

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08/28/99 at 9:35 AM

No.	Doc. I.D.	Document Title
1	SWL-IN-405	pH Electrometric Measurement (Test Codes IN200 & HW200)
1	SWL-IN-406	Determination of Colorimetric Hexavalent Chromium (MT169)
1	SWL-IN-407	Fluoride by Ion Selective Electrode
1	SWL-IN-408	Analysis of Sulfate (Turbidimetric) in Water
1	SWL-IN-409	Analysis of pH Stability
1	SWL-IN-410	Methylene Blue Active Substance (MBAS) in Water
1	SWL-IN-411	Analysis of Silica, Dissolved in Water
1	SWL-IN-412	Nitrate by ISE
1	SWL-IN-413	Analysis of Iodine
1	SWL-IN-414	Analysis of Fluoride (SPADNS – IN130)
1	SWL-IN-415	Biochemical Oxygen Demand in Water
1	SWL-IN-416	Analysis of Turbidity
1	SWL-IN-417	Analysis of Total and Free Chlorine (Residual)
1	SWL-IN-500	Total Alkalinity, Carbonate and Bicarbonate
1	SWL-IN-501	Sulfide
2	SWL-IN-502	Total Hardness: Titrimetric (IN140) and Calculation (IN145)
1	SWL-IN-503	Determination of Total Kjeldahl Nitrogen
1	SWL-IN-504	Ammonia as Nitrogen (Titrimetric with Distillation)
1	SWL-IN-505	Analysis of Inorganic Chloride
1	SWL-IN-506	Acidity (Titrimetric) (IN005)
1	SWL-IN-507	Color-Visual Comparison Method
1	SWL-IN-600	Analysis of Total, Suspended and Dissolved Solids
1	SWL-IN-603	Specific Gravity
1	SWL-IN-604	Determination of Water (Moisture) Content in Solids
1	SWL-IN-609	Analysis of Settleable Solids
1	SWL-IN-700	TCLP/SPLP Non-Volatile Extractions
1	SWL-IN-701	Zero Headspace Extraction for TCLP/SPLP
1	SWL-IN-703	Pensky-Martens Closed Cup Method for Determining Ignitability
1	SWL-IN-705	Analysis of Oil & Grease (Gravimetric)

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08/28/99 at 9:35 AM

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Standard Operating Procedure

Performance and Systems Audits

Document No.: SWL-GA-108

Rev No. / Date: Rev. 4.0 — 02/09/99

APPROVALS	DATES
Procedure Prepared By	Date
Laboratory Manager or Director	Date
Laboratory Safety Officer	Date
Laboratory QA/QC Officer	Date

(Effective Date for is 5 calendar days after the last signature above - QA/QC Officer)

Document Status

[ ] Controlled DC No. _____ Issued to: _____ Date: _____ Initials: _____

[ ] Official Copy OC No.:_____ Issued to: _____ Date: _____ Initials: _____

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ATTACHMENT 2
Logbooks

Department Master Logbook Log
Laboratory Master Logbook Log

Department Master Logbook Log, SWL-GA-101

GENERAL ADMINISTRATIVE

SOP SWL-GA-101, REV. 2.0 FILE ID: GA101A2a.doc

PAGE NUMBER: _____

DEPARTMENT: _____

Book ID Number	SOP Rev.#	Date Log Book Issued	Date Completed	Department Storage Location	Date Transferred to QA Dept.	Initials

SOUTHWEST LABORATORY OF OKLAHOMA, INC.
1700 West Albany • Broken Arrow, OK 74012 • Office (918) 251-2858 • Fax (918) 251-2599

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Laboratory Master Logbook Log, SWL-GA-101

GENERAL ADMINISTRATIVE

SOP SWL-GA-101, REV. 2.0 FILE ID: GA101A2b.doc

PAGE NUMBER: _____

Department (Series)	Book Identification Numbers	Corresponding SOP & Rev. No.	Date Transferred to QA Department	QA Retrieval Location	Date Transferred to Archives	Transfer Confirmation Initials

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ATTACHMENT 3
Formatting Logbook and Forms

Formatting of Logbooks and Forms
Solids Logbook (For An Example)
Fluoride Standard Batch Form (For An Example)
Specific Conductance Analysis Log (For An Example)

A3-1 Formatting of Logbooks and Forms

A3-1-1 Each logbook or form used, that pertains to an SOP shall be a part of each applicable SOP as an appendix or reference and follow the minimum format structure described in this section. That is, if a logbook or form is used to complete or document a procedure, the logbook and/or forms shall be part of its corresponding SOP included in the Attachments Section (Section 8.0 for General or for the O, I, or R series, an Optional Section numbered after the required Section 14.0).

A3-1-2 In addition to the full laboratory name, each page of any logbook shall have a unique identifier number as described below (which includes each page no.) which shall be traceable and recoverable.

A3-1-3 Each logbook shall be velo-bound in a manner to prevent the easy removal of pages.

A3-1-4 Each logbook shall contain at a minimum the following items on each page.

Example of Solids Logbook:

Laboratory Name:	**Southwest Laboratory of Oklahoma**
Book Name:	**Solids Logbook**
Book ID No.:	**IN-600-SldLog-** (syntax as follows:) IN = O Series / Volatiles Dept. 600 = SOP number SldLog = brief description code 3 = book number + page no.
SOP Document #:	**SWL-IN-600**
File ID:	**IN600A1.doc** (syntax as follows) **IN** = Series **600** = SOP# **A** = Attachment as appears in SOP **1** = order as it appears in SOP
SOP revision #:	**Rev. # 3.2**

A3-1-5 Each form shall contain at a minimum the following items on each page.

Example of Fluoride Standard Batch Form:

Laboratory Name:	**Southwest Laboratory of Oklahoma**
Book Name:	**Fluoride Standard Batch Form**
SOP Document #:	**SWL-IN-102**
File ID:	**IN414F1** (syntax as follows) **IN** = Series **414** = SOP # **F** = Form **1** = order as it appears in SOP
SOP revision #:	**Rev. # 2.0**

Typist: LLR

Solids Logbook, SWL-IN-600

Solids Logbook (IN600A1.doc) SOP SWL-IN-600, Rev. 3.2 Verified: ____ Book # IN-600-SldLog- ____

Type of Analysis (circle): TS, TDS, TSS, TVS, TVSS **Date/Time In:** _____ **Date/Time Out:** _____ **Batch ID:** _____

Analyst Signature: _____ **Temp. In:** _____ **Temp. Out:** _____ **Page** _____ **of Book** _____

Sample ID	Description	Dish Weight (B)	Wet Sample Weight	Dried Sample Residual Weight (A)	Dried Sample Residual Weight (D)	Volume (C)	Calculation	Analyst Initials	Date	Time	Page

Calculations: **TS, TDS, TSS = $\frac{(A-B) * 1000}{C}$** **TVS, TVSS = $\frac{(A-D) * 1000}{C}$** **Fixed Solids = $\frac{(D-B) * 1000}{C}$**

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FLUORIDE STANDARD BATCH FORM

INORGANICS DEPARTMENT

FLUORIDE STD. BATCH FORM (IN414F1.DOC) SWL-IN-414, REV. _____ PAGE _____

Analyst: _____ Date: _____ Reagent (Standards) Batch ID: _____

REAGENTS FOR FLUORIDE

REAGENT	*LOT #*
Sulfuric Acid	_____
Silver Sulfate	_____
Anhydrous Sodium Sulfate	_____
SPADNS	_____
Zirconyl Chloride Octahydrate	_____
Hydrochloric Acid	_____
Sodium Arsenite	_____

STANDARD PREPARATION

Stock fluoride solution ID: _____

Standard fluoride solution:

100ml (Stock fluoride solution) ⟹ 1000ml Analyst: _____

NOTE: The above standard was brought to volume with DI water and has a final conc. of **1.00ml = 0.010mg fluoride**

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SPECIFIC CONDUCTANCE ANALYSIS LOG

INORGANICS DEPARTMENT

SPEC. COND. ANALYSIS LOG (IN414F2.DOC) SWL-IN-414, REV. _____ BOOK # IN414-2-___ PAGE _____

Analyst: _____ Date: _____ Prep Batch ID: _____

Start Time: _____ End Time: _____ Fluoride Std: Batch ID: _____

Last Calibration Performed on Book # IN414-2-_____ Page _____

#	SWL ID #	Initial Wt/vol (g) / (ml)	Volume Final (ml)	Absorb. Reading	Final Value mg/L or mg/kg	%R	Comments
1							
2							
3							
4							
5							
6							
7							
8							
9							
10							
11							
12							
13							
14							
15							
16							
17							
18							
19							
20							

REVIEWED BY: _____

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TM

Standard Operating Procedure

Information Systems Quality Assurance Program

Document No.: SWL-GA-102
Rev No. / Date: Rev. 2.0 — 10/05/00

OFFICIAL COPY RED STAMP INDICATES ORIGINAL

APPROVALS	DATES
Procedure Prepared By	10-5-00 Date
Laboratory Manager or Director	10-13-00 Date
Laboratory Safety Officer	10/13/2000 Date
Laboratory QA/QC Officer	10/13/2000 Date

(Effective Date is 5 calendar days after the last signature above - QA/QC Officer)

Document Status

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[✓] Official Copy OC No.: 2431 Issued to: Linda Larsonovich Date: 11/14/00 QA Initials: _____

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Purpose

This document describes policies and procedures for employees of Southwest Laboratory of Oklahoma for the procurement, modification, maintenance, development, testing, acceptance, and usage of computer software and hardware. This program is limited to those software, spreadsheets, macros and hardware that directly affect and manipulate reportable data. These policies and procedures were developed with guidance from EPA's Good Automated Laboratory Practices 1995 edition and the Navy Installation Restoration Laboratory Quality Assurance Guide. The implementation of this program ensures the data produced via electronic processing meets the data quality objectives of the laboratory.

System Organization and Description

The Southwest Laboratory of Oklahoma information system is organized around a central Laboratory Information Management System (LIMS) database augmented by automated data collection and macro programming to assist down loading into the LIMS database and a series of spreadsheet macros and query tools to automate quality control (QC) checking and data reporting to the client. In addition some manual tests require hand calculation and manual entry into the LIMS database.

General Description of the LIMS System

The Southwest Laboratory of Oklahoma LIMS is a distributed database network using Informix and SQL Server as the relation database management tools. Each department has a departmental database connected to the LIMS through a TCP/IP network. A functional diagram of the LIMS is included as Attachment 1.

Main Components of the LIMS System

1. Proposals are tracked using the proposal database.

2. If an award is made, project officers set up the necessary information for the project in the project database.

3. The sample management section of LIMS allows sample receiving to input data concerning the log in of samples, preparation and archiving, and prepare analytical work sheets.

4. Result information is acquired, reviewed, and verified on the separate departmental databases. For volatiles, BNAs, GC, and HPLC data is pulled from the instruments to Windows NT Server where it is verified, reviewed and approved. QC reports are then generated and data is downloaded to the SQL Server database. Sample information is updated on LIMS throughout the process. For metals and inorganics, information is pulled from instruments by the PCs to a Windows NT Server. Then it is reviewed, verified and approved. QC reports are generated and the data is downloaded to the Informix department database servers.

5. The reporting database has two parts; 1) report generation; 2) diskette deliverables. The system offers a wide variety of reporting options: tabular, QC reports, summary reports, etc. Information is pulled from LIMS to the Local PC from which reports and diskettes are generated. Diskette formats include: standard ASCII, Dbase, GISKEY and custom client-specific reports. The exception is CLP diskettes, which are generated from an in-house developed program – GC and GC/MS; Ward - inorganic/metals.

6. QC management contains Windows programs for QC charts for GC/MS, GC, and ICP metals.

QC Reporting

Quality Control Reporting is performed in three levels

The first level of quality control reporting is available to each analyst in the form of batch logs, run logs, calibration curves and control charts. These reports are available to every analyst and department supervisor for all areas of the laboratory.

The second level of quality control reporting is those quality elements contained in the custom data reports to clients. These reports contain the client's quality control samples and those batch samples needed to qualify the client's data set. These reports have various formats as outlined in the following section, but generally consist of tabular and graphical representations of the data.

The third level is the reports and functions available to the QC/QA department to monitor continuing quality of the analytical measurements of the lab. These reports consist of control charts, tabular data by department, and quarterly and yearly summaries.

Data Reporting

There are several main programs used for reporting LIMS data once it has been collected and verified within the LIMS Distributed Databases. The program used depends on the needs of the client. If no diskette deliverable is required the LIMS reporting program can be used to print standard tabular and Prep QC Reports. If data diskettes are required, the data must be imported from the LIMS Databases into the in-house developed reporting program known as "LabMan."

The LabMan program is used by the reporting staff to manage data sets, by Client and Project. All of the data needed for the final diskette deliverable is collected into this project by several methods. Refer to Report Manager Data Flow Diagram included as Attachment 2. Once all of the data has been assembled into the project, the diskette and hard copy can be generated. The hard copy typically consists of standard tabular reports per sample. QC reports for sample prep are also included.

The LIMS reporting program performs some calculations on the data used in the final report. The data reported and calculations performed are based on several reporting options. Currently the Calibration options are not functional.

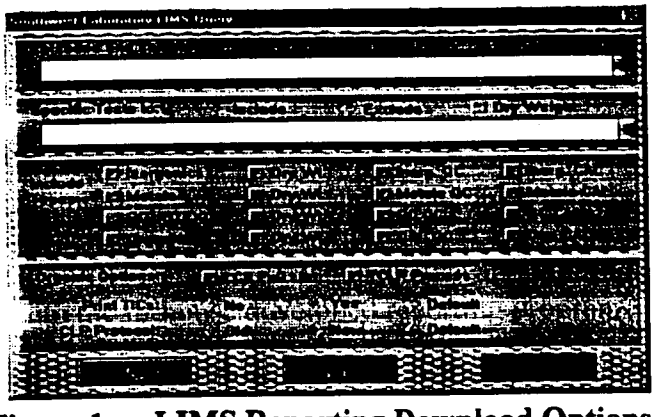


Figure 1 — LIMS Reporting Download Options

This screen is used to query or download specific Prep QC information. From the options the above the query program determines what data to retrieve from the database. Some options determine how the data is calculated. The episode number is used to refer to specific client and project so the correct project database information can be applied. Project information in the projects database is be used to determine the final rounding of numbers and compound lists to be reported.

Listed below are the various types/clients electronic deliverables.

CLP OLM04.2	CLP ILM03.0
CLP OLM03.2	CLP ILM04.0
ERMA	IRDMIS
GIS/Key 1.3,1.5, 2.1	ERPIMS
IRPIMS 2.3, 3.0	Individual Client Requested Formats

Also, a results database and sample review program (CMENU) is available to clients.

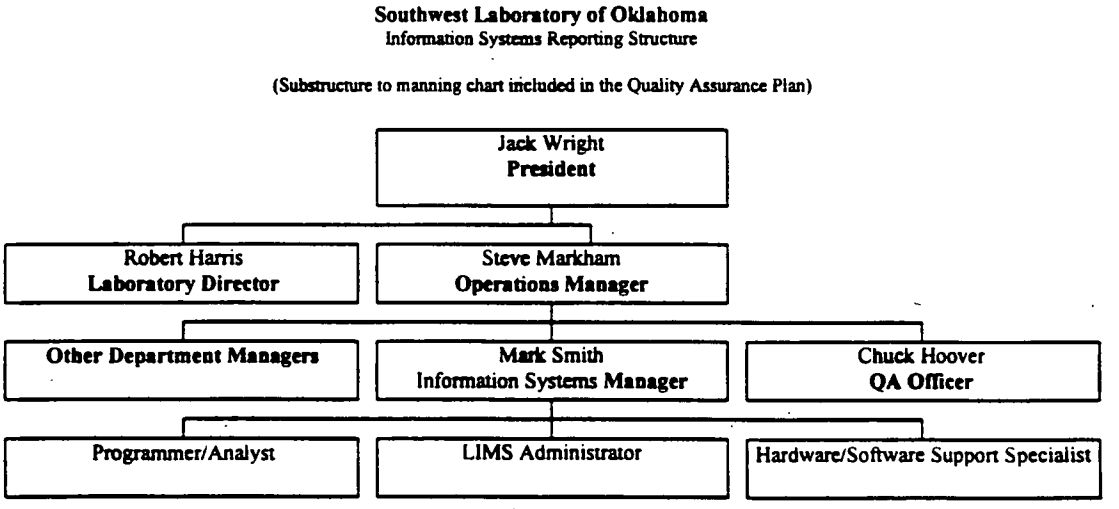
Data Manipulations

Some results are hand calculated and hand entered into the LIMS. The majority of these results are for wet chemistry parameters for which there is no automated instrumentation.

Personnel Responsibilities

Reporting Structure

The following is a reporting structure chart for Southwest Laboratory of Oklahoma Computer Department.



Information Systems Job Descriptions

Job descriptions for the following job titles for the computer department are included in Attachment 4.

- Information Systems Manager
- Programmer/Analyst
- LIMS Administrator
- Hardware/Software Support Specialist

Authorization, Verification and Approvals

Programs

The President. Laboratory Director. and Operations Manager authorize any new programming to be done by the IS staff. with the exception of small spreadsheet and macro programming requests from the laboratory supervisory staff.

All verification of commercial and developed software. spreadsheets. and macros is performed by the department initiating the request and is approved by the department manager.

All validation of commercial and developed software. spreadsheets. and macros. is performed by the QA Officer or designee.

Spreadsheets

For those tasks that involve routine. repetitive. or complex calculations. the use of a spreadsheet can greatly assist in maintaining data integrity. Southwest Laboratory of Oklahoma uses the Excel and Quattro Pro product lines. If a spreadsheet is to be used in the reduction or reporting of data. steps must be taken to assure the spreadsheet is reliable and maintains data integrity. Outlined below are guidelines for the development and acceptance of spreadsheets.

Spreadsheet Development

Most spreadsheets are developed by someone other than computer programming personnel. The use of any spreadsheet, or modification of existing spreadsheets. that would affect the analytical data and reporting of the laboratory, must be documented. verified. validated and approved.

All spreadsheet are designed for ease of use. Cells containing formulas must be locked as not to permit accidental alteration.

Spreadsheet Evaluation

Documentation consists of a transfer of an electronic copy of the spreadsheet. hard copy documentation of the code logic and formulas used to generate the spreadsheet data. a written purpose and requirements for the software, and a Software Programming Request form to be presented to the QA Officer. All validation is performed by the QA Officer or designee. Upon successful completion of the validation process the QA Officer approves the spreadsheet for use in the laboratory and present all documentation and approvals to the LIMS Administrator who assigns a Software Control Number from the Software Control Logbook.

Implementation

The Software Control Number is a two segment number consisting of a serialized identification (ID) and the date of approval. This number is embedded in an unalterable spreadsheet cell in A1

All documentation, electronic copies and verification/approval forms are archived for a period of not less than 10 years.

Updates

All updates shall be handled as for newly developed spreadsheets

Macros

Most macros are developed by someone other than computer programming personnel. The use of any macro, or modification of existing macros, that would affect the analytical data and reporting of the laboratory, must be documented, verified, validated and approved.

All macros are designed for ease of use. Where possible, macros are write protected to prevent accidental changes.

Macro Evaluation

Documentation consists of a transfer of an electronic copy of the macro, hard copy documentation of the code logic and formulas used to generate the macro data, a written purpose and requirements for the software, and a Software Programming Request form to be presented to the QA Officer. All validation is performed by the QA Officer or their designee. Upon successful completion of the validation process the QA Officer approves the macro for use in the laboratory and present all documentation and approvals to the LIMS Administrator who assigns a Software Control Number from the Software Control Logbook.

Implementation

The Software Control Number is a two segment number consisting of a serialized ID and the date of approval. This number is embedded in the remarks coded into the macro.

All documentation, electronic copies and verification/approval forms are archived for a period of not less than 10 years.

Updates

All updates shall be handled as for newly developed macros.

Equipment And Software Purchases

Equipment and software purchases authorization and approval is given by laboratory management. All software and equipment must be maintained, verified and validated as defined in this Program.

Training

If deemed necessary by laboratory management, user training for the new software is performed. Training is conducted according to the Laboratory Training SOP.

Facilities

All facilities for IS computers and equipment are the same as described for the laboratory in the Laboratory Quality Assurance Plan.

Humidity, Temperature Control

All PC and server equipment are room temperature tolerant and therefore are contained in the general environment of the laboratory with the other analytical instrumentation. No special environmental controls are required.

Protection from Electrical Noise & Outages

All LIMS and Windows NT servers are protected from electrical loss and/or surge via individual UPS systems.

Personnel Access

Access to the computer department is the same as defined by the normal laboratory building access restrictions found in the Laboratory QA Plan. PCs are secure through normal laboratory building access restrictions. LIMS servers have been placed in a locked room with access limited to the Computer Department personnel, Laboratory Manager and Laboratory Director.

Disaster Plans

All nightly backups of database systems and NT servers are taken home by the LIMS Administrator.

Equipment Hardware

All PCs are 486 and Pentium Intel based processors and are located throughout the lab. The exception is one 286 used for HPLC pump control. The network type is an Ethernet 10BaseT. The networking protocols used are TCP/IP and Netbios.

Design

All computers are purchased as commercially obtainable industry standard equipment.

Location

Computers are located in all laboratory areas and administrative areas of the laboratory. Relocation of equipment between facilities is controlled through the Equipment Location Transfer Document Form included as Attachment 4.

Maintenance

Maintenance is performed on an as-needed basis. Maintenance requests are communicated through use of the Computer Service Request Form. A hardware maintenance log is maintained, by the Hardware/Software Support Specialist for the main LIMS, Databases, and file servers. The log is kept electronically on the SWLO3 server in the Documents directory under the file name "maintenance log.xls." Please see Attachment 5.

Repeated failures are evaluated by the computer department and reported to laboratory management for need to correct problem.

Security

LIMS servers have been placed in a locked room with access limited to the Data Management Personnel, Laboratory Manager, and Laboratory Director.

PCs are secure through normal laboratory building access restrictions.

Life Cycle

Hardware is upgraded as deemed necessary by laboratory management based on capacity and laboratory technical needs. The Laboratory President or Laboratory Director request and authorize new hardware for all areas of the laboratory.

When a new version or upgrade or modification to computer systems that affect the laboratory reporting data are performed, all outdated and replaced hardware is removed from active use by the lab and equipment is archived, for a period not less than 10 years, to allow for future retrieval and processing of historical data.

Software

Southwest Laboratories of Oklahoma computer department manages all the software used throughout the laboratory with the exception of spreadsheets developed by the different laboratory departments.

The computer department does control release of all software, commercial and in-house, including spreadsheets that directly affect any reportable laboratory data.

A listing of all commercially used software packages is maintained by the LIMS Administrator as part of the Software Control Logbook.

Commercial Data Packages

There are numerous software packages that can assist the user in the compilation, reduction, and reporting of data. It is essential that the software be reviewed and tested to verify that it is reliable and maintains data integrity. Outlined below are guidelines for acceptance of commercially developed software.

If the software performs operations that directly effect the laboratory data, it must be treated and tested as outlined for in-house developed software Maintenance, Verification and Validation, below.

If the software does not pass verification and validation, it must not be used in the laboratory. The results of the test and the problems encountered are to be verified. The software package name and version and the persons testing and the date tested must be documented in the Software Control Logbook.

After the evaluation is complete and the software deemed acceptable, the software may be made available for use in the laboratory. The vendor licensing agreement and copyright laws must be followed. Any changes, patches or updates to the approved program and approved version number must be tested and approved as outlined for in house developed software Maintenance, Verification and Validation, below.

Maintenance

Initiating a Change

There are numerous reasons that warrant a software modification. These reasons include the changing needs of clients or users, fixing errors, implementing enhancements, or increasing the programs performance. Whatever the reason for the modification, great care must be taken so that the modification does not introduce new errors. The guidelines set forth below are meant to inhibit the introduction of new errors into the system.

Software modifications should be initiated through the Software Programming Request form (Attachment 7). Submit the completed form, along with any supporting documentation, to the Information Systems Manager. Supporting documentation includes error logs, reports with incorrect values, diagrams of screen modifications, etc.

Modifications

If possible, modifications are made by the program's developer. If the programmer requires more information than supplied, the programmer is to discuss the problem with the person submitting the request and refer to any other methods, references or documentation as appropriate.

Modifications are made on a development system and tested by the programmer. When the change(s) are made the software is tested as outlined for new programs. If the standard test data set does not include data that tests the modification, the test data must be expanded.

If errors are found, a copy of the error list is to be given to the programmer. The cycle of testing and programming corrections continues until the program is determined to be reliable and maintains data integrity.

The successfully modified version of the program is assigned a new Software Control Number, the associated documents for the new Software Control Number are filed by the LIMS Administrator and the modified software released for use by the laboratory.

If the program is being used in the laboratory, all old revisions must be updated. If necessary, the users manuals are amended to reflect the modification. A copy of the old revision of the software must be retained for a period no shorter than 10 years.

Verification

All new software must be verified. Verification is performed by the individual or department submitting the request. The verifier is responsible to design a test case that exercises the software to its boundaries. The results are compared against results determined by another method (e.g. manual calculations, results from a different program or spreadsheet, etc.). Screen, report, and file formats are checked for correctness. The verifier must complete the Software Programming Request Form included as Attachment 7.

If the software passes the Verification, It must then be Validated. If the software does not perform as expected, the program must be revised and verified.

Validation

Validation of a software package is done by the QA Officer or their designee using at least one complete set of suitable sample data with results known by another method (e.g. manual calculations, results from a different program or spreadsheet, etc.). This sample set is to contain all parameters affected or expected by the software package under test. The QA Officer completes the Software Programming Request Form.

If errors were discovered, the Software Programming Request Form and the test data are given to the programmer. The programmer makes program corrections, and submits the corrected software for re-verification and validation. This cycle of testing and program correction continues until the program is determined to be reliable and maintains data integrity. In the case of commercial software the package must be rejected for use by the laboratory if it fails the validation.

If no errors are discovered, the software package is released for use in the lab. The successfully modified version of the program is assigned a new Software Control Number, the associated documents for the new Software Control Number are filed and the modified software released for use by the laboratory.

If the program is being used in the laboratory, all old revisions are updated. If necessary, the users manuals must be amended to reflect the modification. A copy of the old revision of the software is retained for a period no shorter than 10 years.

Documentation

Software packages covered by this Program and in use by the laboratory are assigned a serial Software Control Number. The Software Control Number is maintained in a Software Control Logbook held by the LIMS Administrator. This logbook lists the control number, software title, version number, date of testing , person testing and approval status ("Passed" or "Failed" to denote status).

All major software systems must have a users manual. These manuals are available from the computer department for all users.

The department supervisors in which spreadsheets or macros are developed and used are responsible for the documentation and testing of the spreadsheets and macros, with the exception of the HP Chemstation macros.

The Information Systems Manager is responsible for documentation and testing of in-house developed programs, including HP Chemstation macros.

In order to facilitate future changes and modifications to all in-house developed programs, macros and spreadsheets, each must be well documented. Documentation is in two forms, program comments and a written explanation of the flow of the program. This explanation includes a listing of the source code files used and their function, a listing of the functions of each source code file and their purpose, and any peculiar problems and how they were solved. This documentation is kept is the software development file cabinet in a folder labeled with the Software Control Number.

Security

The LIMS Administrator maintains the LIMS Access Log which documents the names and types of LIMS access allowed by all staff at the laboratory. The log is an Excel spreadsheet stored in SWLO1:\Robyn\Misc\Security.xls on the (see Attachment 11 – Example).

Life Cycle

Software is upgraded as deemed necessary by laboratory management. The Laboratory President or Laboratory Director request and authorize software development for all laboratory systems. The exception is for spreadsheets and macros that can be requested from the laboratory department supervisors.

All requests for software programming and evaluation are documented on three types of forms:

1. Software Programming Request Form
2. Test Code Addition Request Form
3. Database Modification Request Form

The Software Programming Request form documents needed development or service for software throughout the laboratory. It is completed by the laboratory department personnel and submitted to the Information Systems Manager. The Information Systems Manager is responsible for delegating and completing approved request.

The Test Code Addition Request form (Attachment 9) documents the need of an addition of a test code to the LIMS database. The form is completed by the laboratory departments and submitted to the Information Systems Manager. The IS Manager is responsible for, delegating and completing the Test Code Addition Request.

The Database Modification Request form (Attachment 10) documents the request of a change to the data in the LIMS database. It is completed by the laboratory department personnel and submitted to either the QA Officer, Laboratory Manager, Laboratory Director, or the Laboratory President for approval. The IS Manager is responsible for delegating and completing the Database Modification Request after approval.

In addition to these forms, written memos from the Laboratory President or the Laboratory Manager documents and initiates any in-house or commercial software package development or evaluation.

After initiation of any software programming, review or modification, a Software Control Number is assigned from the Software Control Logbook maintained by the LIMS Administrator, after the program, macro or spreadsheet has been validated and verified as performing properly.

This approved software is then released to the lab. In the case of a program modification or upgrade the IS Manager is responsible to assign personnel to affect the changes throughout the laboratory. The personnel, dates, and extent of the changes shall be documented in the Software Control Logbook for these larger scale upgrades. Smaller releases for specific macros or spreadsheets can be installed by the computer users in the requesting departments.

When a new version or upgrade or modification to computer systems that effect the laboratory reporting data are performed, all outdated and replaced software is removed from active use by the lab and copies are archived, for a period not less than 10 years, for future retrieval and processing of historical data.

Weekly reporting server backups, client data archives, and departmental database backups shall be maintained in the limited access server room. All nightly backups of database systems and NT servers are taken off-site each night by the LIMS Administrator.

Documentation Record Keeping

The original hard copy of this manual is on file in the SOP file cabinet. When a modification is made, the hard copy of the previous original is moved to the historical SOP file cabinet, and the new original is placed in the SOP file cabinet. Updates are given a new revision number and the date is updated to reflect the new installment date. Copies of this manual are available to all computer department, department supervisors and laboratory personnel with authority to develop software, macros and spreadsheets.

Data Handling

All sample data generated by the laboratory must be recorded unaltered from the analytical process and entered into the LIMS as generated.

There are some results that are hand calculated and are hand entered into the LIMS. The majority of these results are for wet chemistry parameters for which there is no automated instrumentation.

The analytical tests that require manual entry are the only processes allowed to hand enter analytical data into the LIMS system. All manual entry type analysis must have an analytical logbook containing all raw data, calculations and final report result for all analysis entered into the LIMS.

Verification of manually entered results into the LIMS is performed by the reporting personnel. Before data is reported, reporting personnel will compare the results in the LIMS with the hardcopy raw data. If no discrepancies are found, the reporter will sign off on the reporting checklist. If discrepancies exist, a non-conformance form is completed and given to the Departmental Supervisor or their designee. The non-conformance form will be signed and return when the data has been corrected. The form will be attached to the reporting checklist.

Editing of any LIMS test results is only be performed by the Laboratory President, Laboratory Director, Department Supervisors, or their designee. All edits of LIMS data is recorded by the LIMS, date, time and ID tagged and the original result are stored and unalterable as a function of the LIMS audit trail.

Data Security

Data security is managed by the laboratory on two levels. The physical operation and continued operation of all computer systems throughout the laboratory is the first assurance of Data Security. The second level of data security is in the use of passwords and other security features in the software to manage access and control, and document access and modifications to the data.

System Security

Southwest Laboratory of Oklahoma has installed uninterrupted power supplies (UPSs) on all computers running Unix or Windows NT systems. These UPSs provide adequate shutdown time in the event of a power failure to these computers.

Passwords

Southwest Laboratory of Oklahoma uses a login/password security systems for it's LIMS, multi-user data systems, and file server access. System passwords are only distributed to those individuals who need that level of access. System passwords are changed as necessary, such as, when an individual with knowledge of the passwords leaves the company.

For LIMS, a password is required to be set, by the user, upon the first login of a new account. For NT servers, a default password is set by the hardware software support personnel. A new password is requested the first time a user logs in. Password privacy and maintenance is the responsibility of the individual user.

Virus Protection

Southwest Laboratory of Oklahoma uses McAfee virus protection software for all computers running Windows. This software automatically scans the system for viruses during the boot-up process. Viruses are also detected (scanned for) when a peripheral device, such as a floppy disk or CD-ROM is accessed.

Southwest Laboratory of Oklahoma policy requires that all diskettes, electronically downloaded files, or software brought into the laboratory be scanned for viruses by the computer department.

File servers shall be scanned on a quarterly basis to assure that they are free from viruses. This is the responsibility of the hardware / software support personnel. Documentation of the virus scan is maintained in the computer maintenance log.

Access

New employees get computer access by request of the department supervisors. The department supervisors request access using the Computer Service Request form. The completed form is submitted to the LIMS Administrator. The LIMS Administrator is responsible for assigning the requested access level, recording the changes in the Access Form, and informing the department supervisors who submitted the request.

Employees' accounts are disabled when they leave. Department supervisors must notify the LIMS Administrator and the IS Manager by using the Computer Service Request form when an employee leaves.

Monitoring

All LIMS and departmental databases are monitored using a Weekly Usage Checklist Form. This monitors file system free space and database free space. The Weekly Usage Checklist Form is included as Attachment 12.

Back-up Frequency

Automated backups are made daily for each Unix and Windows NT server. Tapes are verified for completeness and rotated daily. Backups on LIMS and databases are monitored with a backup log form included as Attachment 13.

Storage Location

All LIMS and Department Database backups shall be stored in the server room. All nightly backups of database systems and NT servers are taken off-site each night by the LIMS Administrator.

Data Reporting

All data reporting requirements are set forth in the Laboratory Quality Assurance Plan. Electronic deliverables are generated by the LIMS and it's application. The electronic deliverables are checked for completeness and accuracy by the laboratory data reporting staff.

There are several main programs used for reporting LIMS data once it has been collected and verified within the LIMS Distributed Databases. The program used depends on the needs of the client. If no diskette deliverable is required, the LIMS reporting program can be used to print standard tabular and Prep QC Reports. If data diskettes are required, the data must be imported from the LIMS Databases into a 'LabMan Project'.

The LabMan program is used by the reporting staff to manage data sets, by Client and Project. All of the data needed for the final diskette deliverable is collected into this project by several methods (refer to Report Manager Data Flow Diagram). Once all of the data has been assembled into the project, the diskette and hard copy can be generated. The hard copy typically consists of standard tabular reports issued per sample. QC reports for sample prep are also included. An error checking routine is available through the LabMan program, in addition to the error checking routines provided through various agencies (e.g.; QCTOOL and IRPTOOLS-PC for AFCEE IRPIMS)

Subcontractors

Subcontractors are used in the computer operations for programming support.

Programmers

All programming subcontractors are under contract to Southwest Laboratory of Oklahoma and as such must adhere to all provisions of the Information Systems Quality Assurance Program and the Laboratory Quality Assurance Plan.

Southwest Laboratory of Oklahoma retains exclusive rights to all programs developed by the subcontractors.

Hardware Support

Sub-contractors for hardware support are not currently used.

Other Vendors

Other vendors may be used by the laboratory to assure the continuing operation of equipment that affects the laboratory data and reporting.

All subcontracts for computer and information system support must also adhere to all provisions of this Information Systems Quality Assurance Program and the Laboratory Quality Assurance Plan.

Supplies and Materials

Quality of Materials

All computer supplies are purchased as industry standard products from well known vendors.

Complaints and Corrective Action

All complaints and corrective actions involving software or electronic data are handled as part of the complaints and corrective action procedures outlined in the laboratory Quality Assurance Plan.

All changes to the laboratory software or electronic data as a result of a customer complaint or corrective action are performed in accordance with this Information Systems Quality Assurance Program

Documentation

The Software Programming Request and Computer Service Request forms document service for software or hardware throughout the laboratory. The forms are completed by the laboratory department personnel and submitted to the IS Manager. The IS Manager is responsible delegating and completing an approved request.

Documentation of any changes made to the laboratory software or electronic data is maintained as outlined in this Information Systems Quality Assurance Program.

Responsibilities

The IS Manager is responsible for completing customer complaints and corrective actions involving the computer department.

The IS Manager is responsible for changes to the laboratory software or electronic data as a result of a customer complaint or corrective action involving the computer department after appropriate approvals.

The Department supervisors are responsible for customer complaints and corrective actions involving spreadsheets developed within their departments.

Resolution

The IS Manager is responsible for the resolution of all customer complaints and corrective actions involving the computer department.

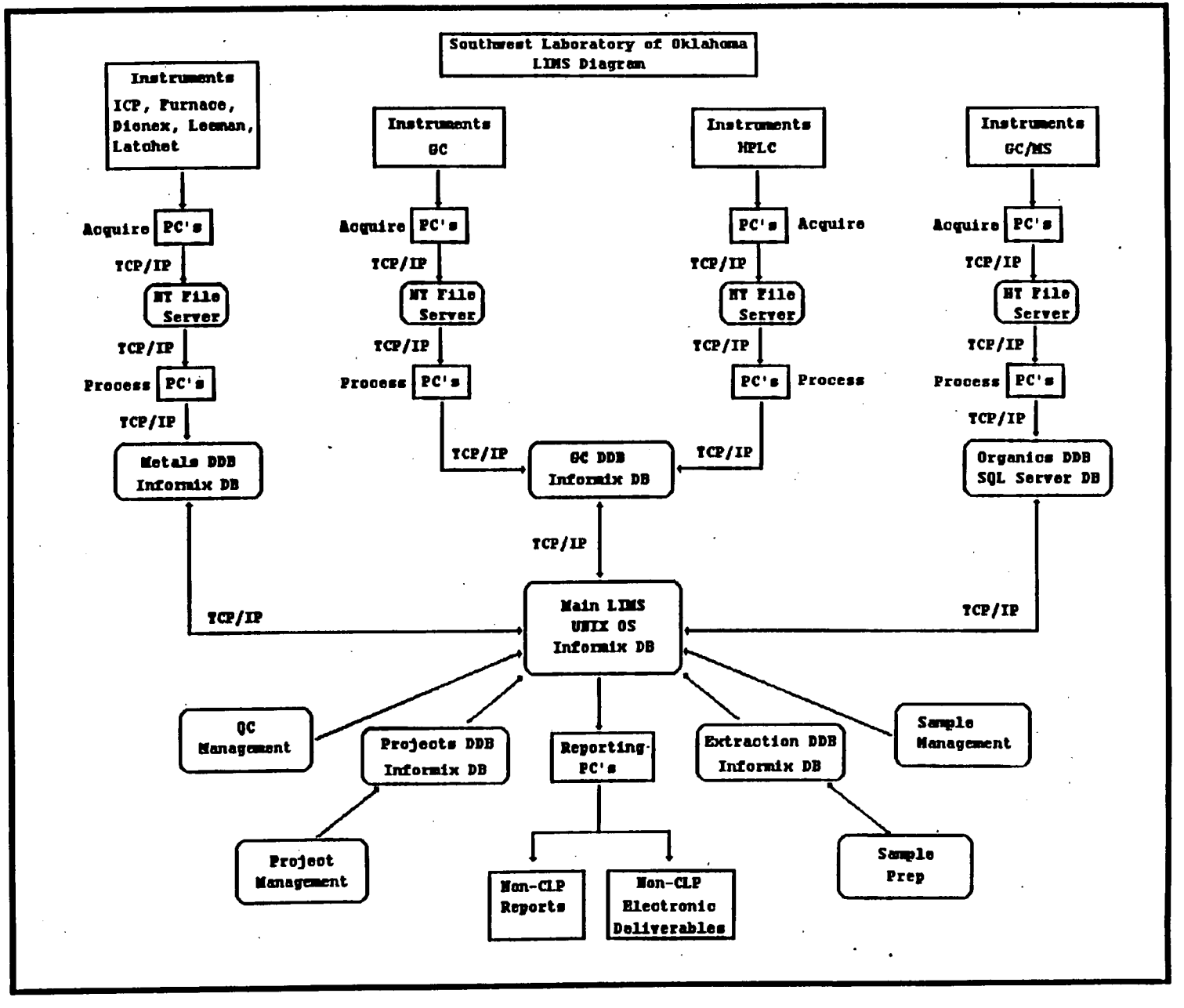
Records

All records associated with the complaint or corrective action must be maintained as outlined in the complaints and corrective action procedures in the Laboratory Quality Assurance Program.

All records of changes to the software or electronic data, all old and replacement versions of the software and the old and replacement electronic data must be retained as outlined in this Information Systems Quality Assurance Program. In all cases, all changes, old and replacement software, old and replacement electronic data is retained for a period of not less than ten (10) years.

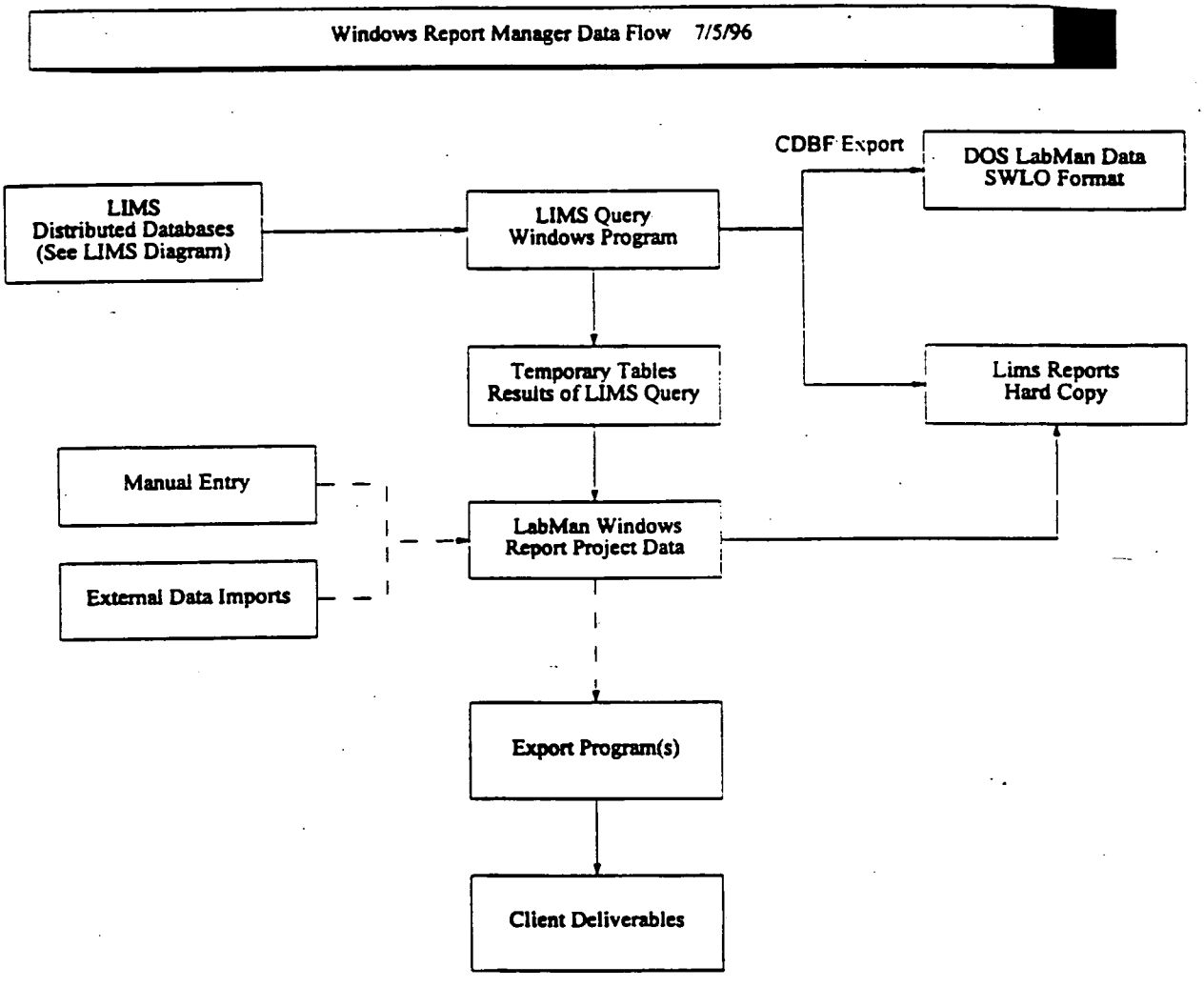
ATTACHMENT 1

LIMS Map



ATTACHMENT 2

Report Manager Data Flow Diagram



Dashed lines indicate incomplete program steps

ATTACHMENT 3

Job Descriptions

Job Descriptions for the following positions follow:

- Computer Services Manager
- Programmer/Analyst
- LIMS Administrator
- Hardware/Software Support Specialist

JOB DESCRIPTION

POSITION TITLE
INFORMATION SYSTEMS MANAGER

POSITION SUMMARY
Develop computerized systems for managing data flow within the laboratory. Monitor and maintain computer hardware. Evaluate new computer hardware/software needs. Oversee the Computer Department's operations and personnel. Work is substantially complex and requires the selection and application of varied guidelines, often covering several functional areas. Independent judgement is needed to identify, select, and apply appropriate procedures.

MAJOR DUTIES AND RESPONSIBILITIES

- Provide technical support to the laboratory personnel. Install software, answer questions, and repair malfunctioning equipment.
- Develop computer software for managing data flow within the laboratory. Evaluate the needs of a particular application and write computer programs to fill the needs. Decide on software design for systems under development.
- Maintain central multi-user computer system. Perform back-ups, keep disk drive cleaned off, and monitor system performance to reduce bottlenecks.
- Install computer hardware as needed for networks involving computer system cabling and software for networks to connect computer to improved data flow.
- Evaluate new computer hardware and software needs. Communicate with users and management about future needs. Keep current on technology.
- Perform other duties as assigned.

SUPERVISION EXERCISED
Supervises the entire Computer Services Department consisting of subordinate professional Programmer/Analyst personnel. Plans and assigns work, and participates in accomplishing work as a hands-on manager. Recommends new hires, promotions, and terminations. Trains and develops staff.

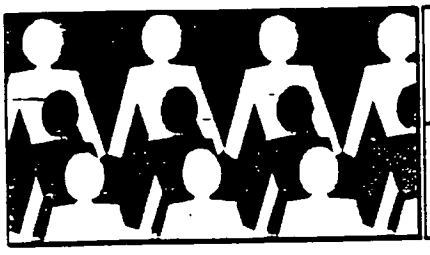
SUPERVISION RECEIVED
Work is occasional reviewed in progress, but is normally reviewed after-the-fact. Guided by established practiced and procedures but exercises independent judgement to achieve predetermined end results.

POSITION REQUIREMENTS

- Bachelor's Degree in Computer Science or Chemistry with a Computer Science minor, and 1 - 3 years of related experience, or an equivalent combination of education and experience.
- Knowledge/experience with the use of
  - Microcomputers and peripherals
  - Electronic test equipment
  - "C" Language Programming
  - UNIX Operating System
  - MS-DOS Operating System
  - FoxBase/DBase/Clipper
  - Hardware Installation

WORKING CONDITIONS

Position requires climbing in order to place cabling in the ceiling for computer connections. Approximately 50% of the workday is spent standing or walking. Position requires the lifting and transporting of Personal Computers and related computer equipment weighing approximately 30 lbs. Demonstrated ability to concentrate on detailed material for long periods of time, and to think abstractly, independently, and creatively.



SOUTHWEST LABORATORY OF OKLAHOMA, INC.
COMPUTER SERVICES DEPARTMENT

JOB DESCRIPTION

POSITION TITLE
PROGRAMMER/ANALYST

POSITION SUMMARY
Design and develop new custom application programs as well as improve existing computer programs. Provide assistance to end-users concerning computer programs. Maintain hardware systems and evaluate new hardware and software products. Work is substantially complex and requires some independent judgement within established guidelines such as setting task priorities, evaluating results and coordinating with others.

MAJOR DUTIES AND RESPONSIBILITIES

- Design and develop new custom application programs to solve data processing problems.
- Improve existing computer programs.
- Provide assistance to end-users to resolve a variety of PC problems.
- Evaluate new hardware and software products to determine if their use could increase efficiency.
- Perform other duties as assigned.

SUPERVISION EXERCISED
None.

SUPERVISION RECEIVED
Supervision of progress and results. Free to determine alternate methods and procedures to achieve predetermined end results.

POSITION REQUIREMENTS

- Bachelor's Degree in Computer Science and 1 - 3 years of related experience, or an equivalent combination of education and experience.
- Knowledge/experience with the use of
  - Microcomputers and peripherals
  - "C" Language Programming
  - MS-DOS
  - FoxBase/DBase/Clipper
  - UNIX Operating System

WORKING CONDITIONS
Approximately 10% of the workday is spent standing or walking. Position may require the lifting and transporting of Personal Computers and related computer equipment weighing approximately 30 lbs. Demonstrated ability to concentrate on detailed material for long periods of time and to think abstractly, independently, and creatively.

SOUTHWEST LABORATORY OF OKLAHOMA, INC.
COMPUTER SERVICES DEPARTMENT

JOB DESCRIPTION

POSITION TITLE
LIMS Administrator

POSITION SUMMARY
Maintain, back up and archive computer network systems. Create, test and document changes to the LIMS systems. Provide assistance to end users for LIMS and other network systems.

Job duties are well defined and guided by established procedures. Position requires an individual with proficient computer skills and demonstrated ability to work effectively with networked computer systems.

MAJOR DUTIES AND RESPONSIBILITIES
- Perform all system backup and archive procedures and check results. Review error reports and messages, and resolve problem situations.
- Facilitate data transfers to and from affiliated laboratories.
- Provide technical support for the LIMS systems. Maintain access for users. Add and delete users as appropriate. Document and test LIMS system changes.
- Solve system user problems and complete service requests from users in all facilities.
- Order, receive and distribute all computer-related supplies.
- Perform other duties as assigned.

SUPERVISION EXERCISED
None.

SUPERVISION RECEIVED
Supervision guided by instructions and established practices and procedures.

POSITION REQUIREMENTS
High school diploma and 1-5 years of related experience; or an equivalent combination of education and experience. Individual should posses proficient computer/database skills and the ability to work effectively with detailed materials.

WORKING CONDITIONS
Position requires long attention spans, meeting deadlines and working with very detailed materials requiring continual and close visual attention. A substantial portion of the day involves computer system monitoring and problem resolution.

SOUTHWEST LABORATORY OF OKLAHOMA, INC.
COMPUTER SERVICES DEPARTMENT

JOB DESCRIPTION

POSITION TITLE
Hardware / Software Support Specialist

POSITION SUMMARY
Maintain, back up and archive computer network systems. Create, test and document changes to the LIMS systems. Provide assistance to end users for LIMS and other network systems.

Job duties are well defined and guided by established procedures. Position requires an individual with proficient computer skills and a demonstrated ability to work effectively with networked computer systems.

MAJOR DUTIES AND RESPONSIBILITIES
- Purchase, install, replace, and repair all personal computer and network server hardware.
- Purchase, install, upgrade, support all PC and network software including DOS, Windows, MS Office, Quattro Pro.
- Provide hardware. Support ethernet and serial network systems including software, wiring, repeaters and hubs.
- Provide support for all printers including PC and network connectivity and repair of printers when necessary.
- Provide phone system support including phone system hardware components and the phone mail system.
- Perform other duties as assigned.

SUPERVISION EXERCISED
None.

SUPERVISION RECEIVED
Supervision guided by instructions and established practices and procedures.

POSITION REQUIREMENTS
High school diploma and 1-5 years of related experience; or an equivalent combination of education and experience. Individual should possess proficient computer hardware/software skills and the ability to work effectively with detailed materials.

WORKING CONDITIONS
Position requires climbing in order to place cabling in the ceiling for computer connections. Approximately 50% of the workday is spent standing or walking. Position requires the lifting and transporting of Personal Computers and related computer equipment weighing approximately 30 – 50 lbs. Must have the ability to concentrate on detailed material for long periods of time and to think abstractly, independently and creatively.

ATTACHMENT 4

Equipment Location Transfer Documentation

EQUIPMENT LOCATION TRANSFER DOCUMENTATION

ACCOUNTING DEPARTMENT

T M

PLEASE FILL OUT ONE FORM PER ITEM OF EQUIPMENT *SWL • AATS • ATAS*

This form must be filled out by the manager of the department who is transferring equipment out to another department or location. This form will facilitate tracking of equipment on hand in each department at each location. **Return completed form to Accounting Department, Broken Arrow location.** Thank you.

MODEL NO: _____ SERIAL NO: _____

VENDOR: _____ DATE OF PURCHASE: _____

INVENTORY CONTROL NO.(I/C): _____ APPROXIMATE VALUE AT TIME OF TRANSFER (IF KNOWN): _____

DESCRIPTION: _____

_____

ORIGINAL LOCATION: _____ DEPARTMENT: _____

NEW LOCATION: _____ DEPARTMENT: _____

DATE TRANSFERRED: _____

NAME OF EMPLOYEE(S) WHO SHIPPED EQUIPMENT FROM ORIGINAL LOCATION: _____

METHOD OF TRANSPORTATION TO NEW LOCATION: _____

NAME OF EMPLOYEE(S) WHO RECEIVED EQUIPMENT AT NEW LOCATION: _____

SIGNATURE OF EMPLOYEE WHO AUTHORIZED TRANSFER: _____

SIGNATURE OF EMPLOYEE WHO PREPARED THIS FORM: _____

ACCOUNTING DEPARTMENT VERIFICATION

EQUIPMENT RECEIVED AT NEW LOCATION OR DEPARTMENT?: _____

VERIFIED BY: _____

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ATTACHMENT 5

Computer Maintenance Log

Hardware Maintenance Log

System Name	Description of Work	Performed By	Date

ATTACHMENT 6

Computer Service Request Form

COMPUTER SERVICE REQUEST

COMPUTER SERVICES DEPARTMENT

Computer Service Request (102-Att8.doc) SWL SOP Rev. # 2.0 Verified: ____ Book #GA-102-Att8

To be completed by the Requesting Individual

NAME:	DATE:
DEPARTMENT:	

REQUEST TYPE: Hardware ☐ Software ☐ Other ☐
EXPLANATION OF REQUEST:

DATE NEEDED BY:	
AUTHORIZATION BY DEPT. MGR.:	DATE:
* AUTHORIZATION BY ADMINISTRATION:	DATE:

To be completed by the computer department

EST. REQUIRED HOURS:	EST. COMPLETION DATE:
BY:	DATE:

PROJECT COMPLETED:

BY:	DATE:

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ATTACHMENT 7

Software Programming Request Form

SOFTWARE PROGRAMMING REQUEST FORM

Software Programming Request Form (102-Att9.doc) SWL SOP Rev. # 2.0 Book #GA-102-Att9

INFORMATION SYSTEMS DEPARTMENT

To be Completed by the Requesting Individual

(Submit initial request along with supporting documentation to the Information Systems Manager. When request is completed, have QC Dept. validate, then submit with verification documentation to the LIMS Administrator.)

Name:	Date:
Department:	Date Needed by:
Authorization by Dept. Manager:	Date:
*Authorization by Administration:	Date:

* Needed only for new program, major enhancement, or immediate attention.

New Program ☐ Enhancement ☐ Program Repair ☐

Explanation of Request:

Explanation of Verification:

Software Control Number:	Program:	
Programmed by:		Date:
Verified by:	Pass: ☐	Date:
Validated by:		Date:

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ATTACHMENT 8

Software Control Logbook Form

Software Control Logsheet for the Computer Services Department

Software Control Logsheet (102-At10.doc) SWL SOP Rev. #: ____ Verified: _____ Book #GA-102-Att10

Department:

Starting Control Number:

Ending Control Number:

Control Number	Title	Version or Revision	Date Tested	Testing Done By	Status	Brief Description

ATTACHMENT 9

Test Code Addition Request Form

Test Code Addition or Modification Request Form

COMPUTER SERVICE DEPARTM

Computer Service Request (102-Att11.doc) SWL SOP Rev. # 1.3 Verified: ____ Book #GA-102-Att11

TEST CODE:		
TEST NAME:		
ABBREVIATED NAME:	METHOD:	
PRICE FOR: Soil: _____	Water: _____	Other: _____
REQUIRES EXTRACTION? ☐ (Y/N)	HOLDING TIME: Soil _____	Water _____
REPORTABLE? ☐ (Y/N)	% MOISTURE? ☐ (Y/N)	

PARAMETERS

NAME	CAS #	TYPE	SPIKE AMT.	UNITS	LIMIT

REASON FOR REQUEST:		
CLIENT:		PROJECT:
FREQUENCY:	DURATION:	EST # SAMPLES:
Requested By:		Date
Approved By:		Date
Completed By:		Date

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ATTACHMENT 10

Database Modification Request Form

DATABASE MODIFICATION REQUEST FORM

COMPUTER SERVICES DEPARTMENT

Computer Service Request (102-Att12.doc) SWL SOP Rev. # 2.0 Verified: ____ Book #GA-102-Att12

DATE:
DESCRIPTION:

REASON:	Logging Error ☐	Other ☐
	Data Entry Error ☐	
	Client Request ☐	

REQUESTED BY:	DEPARTMENT:
APPROVED BY:	TITLE:
COMPLETED BY:	DATE:

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ATTACHMENT 11

Group Access Form

DEFINED EXAMPLE

RANK	USER NAME	LOGIN	DEPARTMENT	MENU	PRG	MENU OPTION
2	Sample Receiving	srlab1	Sample Receiving	0	SEL1	Sample Management
				0	SEL2	Worksheet Options
				0	SEL8	Maintenance
				0	SEL9	System Utilities
				1	Login	Login Samples
				1	Cansam	Cancel Samples
				1	dispose	Sample Archive List
				1	smod	Modify Sample
				2	coc	Internal Chain-0f-Custody Frms
				2	manwrk	Print Login Worksheets
				8	Clients	Client Code Maintenance
				8	clist	Print Client List
				9	word	Run Microsoft WORD

Group Access Code

User/Group Name

System Login

Lab Department

0=Main Menu Item
1=Sub Menu 1
2=Sub Menu 2
(etc.)

Menu Option Titles

SEL1 - Menu Set-1 Program
Login: Sub Menu-1 Program Option
Cansam: Sub Menu-1 Program Option

SEL2 - Menu Set-2 Program
Coc: Sub Menu-2 Program Option
Manwrk: Sub Menu-2 Program Option

The example given above is the Group Access Log (reference page 10, Security) maintained by the LIMS Administrator.

ATTACHMENT 12

Weekly Usage Checklist Form

Weekly DB Checklist

Date: _____

	----- File Sys-----	-----Dbase-----	-----Verified-----
1. BA_LIMS	_____%	_____/347500	_____
2. BR_LIMS	_____%	_____/327865	_____
3. ST_LIMS	_____%	_____/184999	_____
4. gclab	_____%	_____/391362	_____
5. voalab	_____%	_____/851104	_____

Southwest Laboratory of Oklahoma, Inc.

ATTACHMENT 13

System Backup Log Sheet

SYSTEM BACKUP LOGSHEET

SYSTEM	DATE	TIME	BACKUP COMPLETE?	TAPE CHANGED	RERUN	RERUN DATE	RERUN TIME	INIT

SOUTHWEST LABORATORY OF OKLAHOMA, INC. *and*
AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.

TM

Standard Operating Procedure
Personnel Training

Document No.: SWL-GA-104

Rev No. / Date: Rev. 6.0 — 06/02/00

OFFICIAL COPY RED STAMP INDICATES ORIGINAL

APPROVALS	DATES
Chuck Hoover Procedure Prepared By	7/31/00 Date
[illegible] Laboratory Manager or Director	08/02/00 Date
Bryan Fuhs Laboratory Safety Officer	8/02/2000 Date
Chuck Hoover Laboratory QA/QC Officer	7/31/00 Date

(Effective Date for is 5 calendar days after the last signature above - QA/QC Officer)

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PERSONNEL TRAINING

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1.0 PURPOSE AND APPLICATION

1.1 The purpose of this procedure is to standardize and formalize the training and qualification program for laboratory personnel. This procedure is also designed to optimize the time that is available for training and to ensure that the personnel who are performing the procedures have adequate training to achieve quality and compliance.

1.2 This procedure is applicable to Southwest Laboratory's Quality Assurance Manual and all Standard Operating Procedures and methods that affect the operation of equipment, testing procedures, generation of data packages and deliverables, and quality of the data produced by Southwest Laboratory of Oklahoma. Training is required of all staff.

1.3 At a minimum, all laboratory and extra-laboratory staff participating in the generation of analytical data and support function to the generation of analytical data technical work of the laboratory shall be required to take quality assurance training. Additional required training shall be limited to functionally relevant areas. Training in General Safety and Radiation Safety is required for all staff who wish to work in the laboratory areas on an unescorted basis.

1.4 Employees having 6 months or more experience with a specific procedure prior to June 1, 1997 shall be exempt from initial proficiency testing, but will be required to demonstrate annual Recertification. (See Attachment 3, Proficiency Certification.) ALL employees performing their duties prior to June 1, 1997 shall be deemed trained to perform the duties of their position via their previous work experience and the Supervisory Approval Memo (Attachment 9).

2.0 RESPONSIBILITIES

2.1 The Laboratory Director or his/her designee shall designate appropriate assignments for training of personnel.

2.2 Training in the aspects of Radiation Safety is the responsibility of the Radiation Safety Officer. (See Standard Operating Procedure *Radiation Safety*, SWL-RD-141).

2.3 Training in the aspects of general Chemical Health and Safety is the responsibility of the Safety Officer.

2.4 Training in the aspects of Quality Assurance and Quality Control is the responsibility of the Laboratory Quality Assurance Officer.

2.5 Training in the aspects of technical work is the responsibility of the Program Managers and Department Supervisors.

2.6 On-the-job training is the responsibility of the Department Supervisors and Group Leaders.

2.7 Maintaining training records for the laboratory staff will be the responsibility of the Quality Assurance Officer or his/her designee.

2.8 The assigned trainee is responsible for reading, understanding, and complying with the Quality Assurance Plan, appropriate Standard Operating Procedures and reference material(s) applicable for the specific procedures used at Southwest Laboratory of Oklahoma, Inc. The assigned trainee is also responsible for complying with and competently performing the requirements of the Southwest Laboratory of Oklahoma Quality Assurance Plan and Standard Operating Procedures. (Refer to SWL-GA-100).

3.0 **EQUIPMENT** Refer to the listing in the specific Standard Operating Procedures for the equipment that is required.

4.0 **PRECAUTIONS.** Refer to the listing in the specific Standard Operating Procedures for any precautions that may be required.

5.0 **PROCEDURE**

5.1 Initial Training

5.1.1 Training shall consist of a combination of formal sessions and on-the-job training.

5.1.1.1 Formal sessions shall include: Introduction to Southwest Laboratory, Confidentiality and Quality Assurance Orientation, Hazardous Materials and MSDS Orientation, Emergency Shower and Eyewash Locations and Use, Fire Extinguisher Location and Operation, Basic Laboratory Safety and Radiation Safety Orientation and Introduction to Standard Operating Procedures. Those employees who will work in the "Radioactive Materials Control Area" will have further Radiation Safety training as per SWL-RD-141.

5.1.1.2 On-the-job training shall be provided to individuals in the technical tasks to be performed.

5.1.2 A record of the training given shall be documented for each of the steps of the training.

5.1.2.1 "General Orientation and Safety Orientation for New Employees and Temporary Employees", form GA104A1, (Attachment 1). The responsible authority as listed in Section 2.0 shall record satisfactory completion of this part of training.

5.1.2.2 Trainee review and understanding of relevant Standard Operating Procedures shall be documented in form GA104A2 "Document Review and Understanding", (Attachment 2).

5.1.2.3 Initial proficiency certification and recertification shall be documented on form GA104A3 "Demonstration of Capability", (Attachment 3), and is used to document an employee's proficiency at a task.

5.2 New Employees

5.2.1 All new laboratory personnel shall receive initial training in the areas outlined in Section 5.1.1.1 and indicate they have received and understood the training by signing form GA104A1 "General Quality Assurance and Safety Training for New Employees and/or Temporary Workers (Attachment 1).

5.2.2 Supervisors of new employees are required to fill out a "New Employee Training Plan" GA104A7 (see Attachment 7), which will be updated throughout the first year of employment by both supervisor and QA.

New employees are not allowed to work without a training plan filled out for the appropriate time period.

NOTE: If it is determined that an employee doesn't have the appropriate training documented for his/her job function, a stop work order will be issued until the supervisor has completed the necessary training.

5.2.3 The new employee will be given the SOP for the method or procedure they are expected to perform. Upon successful completion of initial training, the employee will be assigned to an apprenticeship with a qualified chemist. After receiving on-the-job training the employee will sign form GA104A2. "Document Review and Understanding", (See Attachment 2) indicating they have received and understood the training for the method or procedure.

5.2.4 The new employee will then perform the method or procedure under direct supervision of the qualified chemist. Only the qualified chemist may report the data after careful review. The new employee will not be permitted to perform independent work at a task until satisfactory performance has been demonstrated.

5.2.5 New personnel may be allowed to perform independent work immediately upon employment if in the judgment of the Program Manager they are qualified by education and/or experience to do so for a particular task, are monitored, and pass an appropriate Initial Demonstration of Capability test. The basis for such a determination by the Program Manager shall be documented and filed in individual training files with a IDC Certification Statement (See Attachment 3).

5.2.6 New managers above the level of supervisor will be trained by other managers to ensure the policies, practices, and current quality system operations are understood. This ensures that the system aspects of the organization remain in tact. It also ensures new managers understand the process for operations improvement.

5.3 Initial Demonstration of Capability (IDC)

5.3.1 Before any results are reported using a particular method the employee shall extract / digest / distill / &/or analyze at least four consecutive matrix spikes prepared from a secondary source (or in the case of 8280 Dioxin from another lot number) from the standards used for the initial calibration. Precision & Accuracy

measurements from the four MS aliquots are compared to method or laboratory (blue book) in-house limits to determine acceptable performance.

5.3.2 If unacceptable results are obtained, repeat the test for all parameters that failed to meet criteria.

5.3.3 Once the Department Supervisor or Group Leader deems the employee trained in their task they will notify complete a IDC summary and submit it (along with the raw data) to the QA Department which then completes the NELAP IDC Certification Statement for inclusion into the employee's training file.

5.3.4 For analytes that do not lend themselves to spikes.(e.g. TSS) the IDC can be performed using QC samples from a NIST approved Vendor. For other analytes (e.g. Paint Filter Liquids Test) in-house IDCs can be developed. The QA Manager will submit a Internal Blind Proficiency Test Sample (PTS) to be analyzed by the employee. Based on the results of the PTS the following will occur:

5.3.4.1 If the results of the PTS are within the acceptance limits developed from peer group performance or supplied by the manufacturer, the QA Manager will judge the employee as proficient and a IDC Certificate Statement will be placed in the employees training file. The employee may now perform the method and report results without direct supervision.

5.3.4.2 If the results of the PTS, or selected analytes from a multi-analyte list, are unacceptable the QA Manager will submit a corrective action form to the Department Manager. The cause for failure will be determined, corrected and additional training provided if needed. The QA Manager will submit another blind PTS for the employee as follow-up. No data may be reported by this employee without direct supervision, until a PTS is acceptable.

5.4 Initial Proficiency Demonstration by Employees not Directly Involved in Analytical Data Generation.

5.4.1 Initial proficiency by direct observation of competent performance by the Supervisor accompanied by demonstration of technical understanding of the task to the Supervisor.

5.4.2 Once the Supervisor deems the employee proficient they will notify the QA Manager in writing, who will then place a IDC Certificate Statement in the employees training file.

5.5 Retraining

5.5.1 All technical staff shall be required to attend a training refresher session in the areas of Chemical Health and Safety, and Quality Assurance. Ongoing technical training will be the responsibility of the department supervisors to ensure employees receive the necessary training to perform their tasks properly.

5.5.2 Personnel shall be re-certified annually in the tasks for which they have been qualified. Recertification shall be documented on the employee's Proficiency Certification Forms. Continued proficiency must be demonstrated for each method through the analysis of either a single blind PTS or external proficiency programs. Results for re-certification must be within the control limits supplied.

5.5.3 The Department Supervisors may require formal training for SOPs when significant modifications are made.

5.6 IDC Certifications

5.6.1 The supervisor shall complete a Precision & Accuracy Summary Form for each employee for tasks in which the employee has been qualified.

5.6.2 The QA Manager or designee shall review the P& A Summary, sign the certification approval, and assign the certification expiration date. The certification expiration date shall be one year from the initial certification.

5.6.3 The supervisor shall document the Recertification of employees on or before the certification expiration date. One year from the Recertification date then becomes the new expiration date.

5.6.4 Re-certification shall include the date re-certified, the basis for re-certification and approval of the QA Manager.

NOTE: An employee who is determined not re-certified (having an expired certification) will be issued a stop work order until the re-certification documentation is completed by his/her supervisor.

6.0 RECORDS

6.1 Form GA104A1 "General Quality Assurance and Safety Training for New Employees and/or Temporary Workers" shall be retained in the training records section of the QA file as a permanent record (see Attachment 1).

6.2 Form GA104A2 "Document Review and Understanding" will be retained in the personnel records section of the QA file as a permanent record (see Attachment 2).

6.3 Form GA104A3 "IDC Certification Statement" shall be retained in the personnel records section of the QA file as a permanent record (see Attachment 3).

6.4 "Code of Business Conduct" adherence agreement shall be retained in the personnel records section of the QA file as a permanent record (see Attachment 4).

6.5 "Confidential Information Agreement" shall be retained in the personnel records section of the QA file as a permanent record (see Attachment 5).

6.6 "New Employee Training Plan" (see Attachment 7).

6.7 Employee Training Summary — This checklist shall be retained in the personnel records section of the QA file as a permanent record (see Attachment 8).

7.0 REFERENCES

7.1 Naval Facilities Engineering Service Center. Interim Guidance Document. "Navy Installation Restoration Laboratory Quality Assurance Guide. February 1996"

7.2 National Environmental Laboratory Accreditation Conference. "Quality Systems", Appendix C. July 1. 1999.

8.0 DEFINITIONS

Terminology

Training:..... The process whereby employees are instructed how to perform their duties.

Initial Demonstration of Capability: The procedure to establish the ability to generate acceptable accuracy and precision, and statistical evaluation of the results in comparison to method criteria and/or in-house limits..

Proficiency Test Sample ..... A sample, the composition of which is unknown to the analyst and is provided to test whether the analyst can produce analytical results within specified performance limits.

Technical Analyst: ..... The designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent Quality Controls to meet the required level of quality.

SOPs:..... (Standard Operating Procedures). Written documents which details the method of an operation, analysis, or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks.

Quality Assurance Plan:..... A document stating the quality policy, quality system, and quality practices of an organization.

9.0 ATTACHMENTS

9.1 Attachment 1: Quality Assurance and Safety Training for New Employees and/or Temporary Workers.

9.2 Attachment 2: Document Review and Understanding.

9.3 Attachment 3: Certification Statement.

9.4 Attachment 4: Code of Business Conduct.

9.5 Attachment 5: Confidential Information Agreement.

9.6 Attachment 6: Sample of Laboratory Job Description.

9.7 Attachment 7: New Employee Training Plan.

9.8 Attachment 8: Sample of Employee Training Summary.

9.9 Attachment 9: Supervisory Approval Memo

ATTACHMENT 1

General Quality Assurance and Safety Training For New Employees and/or Temporary Workers

GENERAL QUALITY ASSURANCE AND SAFETY TRAINING FOR NEW EMPLOYEES AND/OR TEMPORARY WORKERS, SWL-GA-104

Personnel Training Form (GA104A1) SOP SWL-GA-104 Rev. # 4.0

Trainee: _____ **Employment Date:** _____

I certify by my signatures below that I have received the orientation indicated.

1) *Introduction to Southwest Laboratory* *Date:* _____

Trainer: _____ Trainee Signature: _____

2) *Quality Assurance: QA Manual. Rev. No.:* __.__ — __/__/__ *Date:* _____

Trainer: _____ Trainee Signature: _____

3) *Safety: Laboratory Safety Plan. Rev. No.:* __.__ — __/__/__ *Date:* _____

A) Hazardous Materials and MSDS Orientation ..... ❑
B) Emergency Shower and Eyewash Locations and Use..... ❑
C) Fire Extinguisher Locations and Operation ..... ❑
D) Basic Laboratory Safety..... ❑
E) Radiation Safety ..... ❑

Trainer: _____ Trainee Signature: _____

QA Files: Initials: _____ Date: _____ Verified By: _____ Date: _____

ATTACHMENT 2

Document Review and Understanding

DOCUMENT REVIEW AND UNDERSTANDING.

SWL-GA-104

Personnel Training Form (GA104A2) SOP SWL-GA-104 - Rev. # 4.0

Trainer: _____ Training Date: _____

Series Document ID: SWL-__ __-__ __ __ **Revision Number: __.__ __/__/__**

Training Subject: _____

Training Materials: _____

Applicable Document(s): _____

The undersigned have read the above referenced Document and understand it's contents and will comply with the procedure as written. Any deficiencies to the document will be appropriately documented and approved by the supervisor.

and / or

The undersigned certify that they have received training on the above mentioned subject and understand the training given.

Print Name	*Signature*	Date

QA Files: Initials: _____ Date: _____ Verified By: _____ Date: _____

ATTACHMENT 3

Proficiency Certification

Demonstration of Capability
Certification Statement

Date:
Laboratory Name:
Laboratory Address:
Analyst(s) Name(s):

Page __of __

Matrix:
(examples: laboratory pure water, soil, air, solid, biological tissue)
Method number, SOP#, Rev#, and Analyte, or Class of Analytes or Measured Parameters
(examples: barium by 200.7, trace metals by 6010, benzene by 8021, etc.)

We, the undersigned, CERTIFY that:

1. The analysts identified above, using the cited test method(s), which is in use at this facility for the analyses of samples under the National Environmental Laboratory Accreditation Program, have met the Demonstration of Capability.

2. The test method(s) was performed by the analyst(s) identified on this certification.

3. A copy of the test method(s) and the laboratory-specific SOPs are available for all personnel on-site.

4. The data associated with the demonstration capability are true, accurate, complete and self-explanatory (1).

5. All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at the facility, and that the associated information is well organized and available for review by authorized assessors.

Technical Director's Name and Title	Signature	Date
Quality Assurance Officer's Name	Signature	Date

ATTACHMENT 4

Code of Business Conduct

Southwest Laboratory of Oklahoma, Inc.

Jack Wright
President

December 3, 1995

To: All Employees

A major factor in the continuing growth and success of Southwest Laboratory of Oklahoma, Inc. and its affiliated Companies (SWL) is the standard of personal and professional integrity with which its men and women conduct themselves.

Policies and standard operating procedures have been in place for some time governing the way in which the Company conducts its business. In addition to these policies and standard operating procedures, the Company is adopting a Code of Business Conduct, a written guide to assist SWL employees in understanding the principals of conduct that must be adhered to in order to fulfill the legal, moral and ethical obligations that each assumes as an employee.

The Company is committed to conducting its business in accordance with the Code of Business Conduct and established policies and standard operating procedures. All employees are expected to conduct their activities in a manner that will promote the observance of these principals and guidelines.

Very truly yours,

Jack Wright

Southwest Laboratory of Oklahoma, Inc.

Code of Business Conduct

Introduction

The purpose of this code is to state the principles of business ethics that the Company expects all employees to follow. The Code goes on further to state specific guidelines for conduct in situations that affect employees.

These principles and guidelines are to be strictly adhered to at all times and under all circumstances. Anyone who does not adhere to this Code is acting outside the scope of his or her employment. Additionally, conduct that does not comply with the provisions of the Code may well constitute a violation of one or more criminal laws.

The following principles and guidelines are applicable to all employees of Southwest Laboratory of Oklahoma and its affiliated Companies ("The Company"). Violations by any employee will result in disciplinary action, including, in proper cases, discharge from employment.

Compliance With All Laws

The Company is committed to being a good corporate citizen of all states in which it does business. Because of this commitment, it is the policy of the Company to comply in all respects with all laws and regulations that are applicable to its business at all government levels in the United States.

The laws and regulations of the states in which the Company does business and the laws and regulations of the United States form the framework around which the Company's operations are built. To comply in all respects with both the spirit and the letter of those laws will best serve the interests of the Company and its employees.

Ethical business conduct should normally exist at a level well above any minimum required by law. The Company expects its employees to deal fairly with all persons with whom the Company does business and to maintain the Company's reputation for integrity in all its business dealings.

Employees should make the Company's legal compliance policy known to all agents and contractors of the Company and inform such persons that the Company expects them likewise to adhere to this policy.

Laboratory Analysis And Test Results

It is the policy of the Company to produce accurate analytical test results. It is the responsibility of all employees to promptly report any information regarding improper performance of analytical testing or misrepresentation of analytical data.

Examples of improper performance of analytical testing or misrepresentation of analytical data include:

1. altering any analytical instrument, computer or clock;
2. altering the contents of any logbook or data sheet;
3. falsifying analyst identity;
4. preparation of data packages that do not faithfully reflect actual analytical data in logbooks or data sheets;
5. calibration techniques at variance with contract requirements, SOP's, industry standards or regulations;
6. any misrepresentation of data or events as they actually occur in the course of data generation, review or reporting;
7. any other improper activity that could result in improper performance of analytical testing or misrepresentation of analytical data.

Any employee having knowledge of any act or circumstance that is prohibited by this policy shall immediately report the matter to a member of the Company's management.

Conflict Of Interest

All employees of the Company have a primary business responsibility to the Company and are expected to avoid any activity that may interfere, or have the appearance of interfering with the performance of this responsibility.

The following will serve as a guide to the circumstances or types of activities that could cause conflicts and therefore should be fully reported to the Company.

1. Disclosure or use by any employee of information that is confidential, proprietary or privileged for the benefit or gain of the employee or any other person. It is the policy of the Company to require employees to sign a written agreement prohibiting unauthorized disclosure of confidential information and misappropriation of the Company's intellectual property.

2. Ownership by an employee or a close relative or associate of a two percent or more financial interest in any enterprise that does business with or is a competitor of the Company.

3. Participation in any outside activity that competes directly or indirectly with the Company or that interferes or has the appearance of interfering with the performance of the employees duties to the Company.

4. Service as a director, consultant or employee of an enterprise that conducts or seeks to conduct business with the Company.

5. Acceptance by an employee or a close relative or an associate of gifts of a size that may tend to influence business decisions or compromise independent judgment. This includes loans, excessive entertainment, or other favors from any individual, enterprise or organization that does, or is seeking to do business with, or is a competitor of the Company.

Failure by an employee to cease and desist any activity that, in the opinion of management, results in a conflict of interest, may result in discharge from employment.

Proper Recording Of Funds, Assets, Receipts and Disbursements

All funds, assets, receipts and disbursements of the Company shall be properly recorded on the books of the Company. To assure that this policy is implemented, it is specifically understood that:

1. No funds or accounts shall be established or maintained for purposes that are not fully and accurately reflected on the books and records of the Company.

2. No funds or other assets shall be received or disbursed without being fully and accurately reflected on the books and records of the Company.

3. No false, fictitious or intentionally misleading entries shall be made on the books or records of the Company and no false or misleading reports pertaining to the Company or its operations shall be issued.

Environmental And Safety Policy

The Company has long been committed to the goal of safe, efficient and environmentally sound business practices and operations and has been supportive of endeavors aimed at preserving our environmental heritage. The Company is committed to complying with all applicable laws and regulations relating to protection of the environment and the maintenance of a safe workplace. These laws and regulations are diverse and far reaching and any violation of them can produce severe consequences not only for the Company but for each employee involved in the violation.

The Company endeavors to maintain a safe workplace for the safety of its employees, customers and the general public. Each employee is required to use such safety equipment as may be required by law, regulation or Company policy. Employees are also required to

use all reasonable efforts to maintain the work area in such condition as will not pose a safety hazard for themselves or others. Employees are encouraged to identify ways to improve safety and to bring them to the attention of their supervisors.

Human Resources

The Company recognizes that its greatest asset is its human resources and realizes the proper utilization and development of this asset is the key to continued success. The Company intends to operate under sound personnel policies and to apply an equitable standard of fair treatment to all its employees.

In order to fully utilize these human resources, it is the Company's practice to:

1. Select and place employees based on their qualifications and without discrimination in terms of race, color, religion, national origin, sex, age or disability.

2. Provide a healthful and safe work environment in which self-development and the broadening of skills are encouraged, free from intimidating conduct.

3. Encourage and facilitate promotion and transfer from within the Company whenever possible.

4. Encourage employees to express freely their ideas and suggestions concerning the Company, its operations and activities.

Violations

Any employee who becomes aware of any conduct he or she believes violates the letter or spirit of this Code should report this condition to his or her supervisor, or to the Director of Human Resources, or to any member of management of the Company.

Southwest Laboratory of Oklahoma, Inc.

Code of Business Conduct

I have received the Company's Code of Business Conduct and have had an opportunity to review the Code and ask questions concerning the provisions of the Code.

I affirm the provisions of the Code and agree to adhere to the principles of conduct and guidelines therein.

As a Southwest Laboratory employee or as an employee of an affiliated Company, I understand that I have a responsibility to report to management any violation of the letter or the spirit of the Code of Business Conduct.

Employee Signature: _____ Date: _____

ATTACHMENT 5

Confidential Information Agreement

Confidential Information Agreement

In consideration of my employment, or the continuation of same by Southwest Laboratory of Oklahoma, Inc. and its affiliated Companies, hereinafter collectively referred to as the "Company", I hereby agree as follows:

I will not without first obtaining the Company's written consent disclose to others, or use (except in performance of my duties for the Company), either during or after my employment, any information or data which pertains to the Company's business or interests and which is not freely available to persons not employed by the Company. Upon termination of my employment I will promptly turn over to the Company all then existing documents and copies thereof made or acquired by me during my employment which contain any such information or data.

Confidential information and trade secrets include, but are not limited to: customer or client lists; price lists; marketing and sales strategies and procedures; business plans and systems; quality control procedures and systems; special projects and technological research, including projects, research and reports for any government entity or client; client's plans and processes; client's manner of operation; trade secret's of clients; client's data; any other records, data, files, drawing, inventions, discoveries, applications or processes which are not freely available to persons not employed by the Company.

I acknowledge that a remedy at law for any breach by me of any of the provisions of the confidentiality agreement will be inadequate and I hereby agree that the Company shall be entitled, where appropriate, to specific performance and/or injunctive relief in case of any breach thereof.

This agreement is not an employment contract and does not give any employee any right to be retained in the service of the Company.

In witness whereof, I have executed this agreement, intending to be legally bound, this _____ day of _____, 19_____

_____ _____

Printed Name Signature

ATTACHMENT 6

Sample of Laboratory Job Description

POSITION TITLE
GC/MS ANALYST

POSITION SUMMARY
Conduct organic analysis of Semi-Volatile and Volatile environmental samples using combined Gas Chromatography/.Mass Spectrometry (GC/MS) following EPA Control Laboratory Program (CLP) protocol. Performs troubleshooting and routine and preventative maintenance on instrumentation. Work is moderately complex and guided by well defined procedures. Performs a variety of tasks that are structured to provide experience and familiarization with EPA protocols and methods.

MAJOR DUTIES & RESPONSIBILITIES
- Ensure that GC/MS is functioning properly by following specified protocols which contain numerous QA/QC checks.
- Review "Holding time" data and obtain samples in appropriate chronological order for analysis. Screen samples and prepare for analysis.
- Analyze samples on GC/MS following specified protocols and previously determined computer format.
- Check data from runs to ensure it meets protocol requirements.
- Assemble initial reports when run is complete. Enter run numbers and dates and forward to Data Clerks.
- Prepare internal standards and calibration standards according to contract specifications.
- Order supplies as needed to run GC/MS instrument.
- Perform instrument maintenance as needed to ensure instruments is operating at maximum efficiency.
- Perform other duties as assigned.

SUPERVISION EXERCISED- N/A

SUPERVISION RECEIVED
Supervision of progress and results. Work is guided by detailed, well-established procedures.

POSITION REQUIREMENTS
- Bachelor's Degree in Chemistry or related science and 0-2 years of related experience; or an equivalent combination of education and experience.
- Knowledge of: GC/MS
  Tekmar (Purge & Trap)
  Computer Terminal/System

WORKING ENVIRONMENT
Standard laboratory environment. Physical requirements are minimal. Requires close attention to detail so as not to exceed "Holding Times" and costly penalties.

ATTACHMENT 7

New Employee Training Plan

NEW EMPLOYEE TRAINING PLAN

SWL-GA-104 REV.4.0 GA104A7

EMPLOYEE: _____	DATE OF EMPLOYMENT: _____
POSITION: _____	SUPERVISOR: _____

1st MONTH

TARGET DATE:

SOP	SOP TITLE	DEMONSTRATION OF PROFICIENCY
GA-100	LABORATORY QUALITY ASSURANCE PLAN	N/A
GA-104	PERSONNEL TRAINING	N/A
GA-111	LABORATORY SAFETY PLAN	N/A
GA-123	HAZARDOUS MATERIALS CONTINGENCY PL	N/A

TRAINING COMPLETED ? Y / N	SUPERVISOR SIGNATURE:	QA SIGNATURE:

1st QTR

TARGET DATE:

SOP	SOP TITLE	DEMONSTRATION OF PROFICIENCY

TRAINING COMPLETED ? Y / N	SUPERVISOR SIGNATURE:	QA SIGNATURE:

6 MONTHS

TARGET DATE:

SOP	SOP TITLE	DEMONSTRATION OF PROFICIENCY

TRAINING COMPLETED ? Y / N	SUPERVISOR SIGNATURE:	QA SIGNATURE:

1 YEAR

TARGET DATE:

SOP	SOP TITLE	DEMONSTRATION OF PROFICIENCY

TRAINING COMPLETED ? Y / N	SUPERVISOR SIGNATURE:	QA SIGNATURE:

ATTACHMENT 8

Sample of Employee Training Summary

Southwest Laboratory of Oklahoma, Inc.

AIR & DIOXIN DEPARTMENT

ANALYST NAME:		Lavant Stringfellow	Title:		Technical	Director
SOP SERIES	ISSUE DATE	METHODOLOGY:	CURRENT REVISION	SOP REV.	PROFIC. DEMO	DATE APP.
[illegible]	[illegible]	Laboratory Quality Assurance Manual	[illegible]	[illegible]	N/A	[illegible]
[illegible]	[illegible]	Preparation Review Revision of Control of Procedure [illegible]	[illegible]	[illegible]	N/A	13-Oct-97
[illegible]	7/31/97	Personnel Training	[illegible]	[illegible]	N/A	14-May-97
[illegible]	7/31/97	Code of Business Conduct / Confidential Information Agreement	[illegible]	[illegible]	N/A	[illegible]-Jul-98
SA-105	6/8/97	Corrective Action	[illegible]	[illegible]	N/A	28-May-97
SA-107	9/30/97	Procurement of Materials & Equipment	[illegible]	[illegible]	N/A	
SA-108	9/16/97	Traceability of Standards	[illegible]	[illegible]	N/A	23-May-97
SA-110	9/17/97	Laboratory Documentation of Sample Custody	[illegible]	[illegible]	N/A	28-May-97
SA-111	6/19/97	Laboratory Safety Plan	[illegible]	[illegible]	N/A	2-Oct-97
SA-112	3/4/98	Control Charts	[illegible]	[illegible]	N/A	3-Oct-97
SA-113	12/1/97	Definition & Determination of Method Detection Limits	[illegible]	[illegible]	N/A	
SA-114	6/6/98	Hazardous Waste Management Plan	[illegible]	[illegible]	N/A	28-May-97
SA-123	6/20/98	Hazardous Materials/Comm. Contingency Plan	[illegible]	[illegible]	N/A	
SA-124	7/24/96	LMS Multiple Test Code Procedure	[illegible]		N/A	21-Jul-98
SA-127	5/14/97	Preventive Maintenance	[illegible]	[illegible]	N/A	
SA-128	10/13/97	Maintenance of "DONE" Logbooks	[illegible]	[illegible]	N/A	
SAI CA-100	12/12/96	Tenax & Tenax-Charcoal Tubes (Preparation)	[illegible]	[illegible]	N/A	
SA-101	2/21/97	Volatile Organic Compounds in Air by MS (MS-401)	[illegible]	[illegible]	N/A	
OA-102	2/25/97	Volatile Organics in Air by GC/MS (MS-414)	[illegible]	[illegible]	N/A	
OA-103	6/13/96	Mod. Method TO-3 Deter. Of Volatiles in Air by GC/MS	[illegible]	[illegible]	N/A	
OA-104	1/14/98	Air Logbook Maintenance	[illegible]	[illegible]	N/A	
OA-105	10/3/97	Air Standards Traceability	[illegible]	[illegible]	N/A	
ODI OD-102	10/15/97	Dioxin Logbook Maintenance	[illegible]	[illegible]	N/A	
OD-103	8/14/97	Dioxin Data Review	[illegible]	[illegible]	N/A	
OD-109	8/22/97	Dioxin Standard Traceability	[illegible]	[illegible]	N/A	
OD-202	2/17/98	Polychlorinated Dibenzo-p-Dioxins-8280A (MS-750)	[illegible]	[illegible]	N/A	22-Jan-98
OD-203	5/7/98	Polychlor.Dibenzo-p-Dioxins-Dibenzofurans (EPA DFLM0.10)-MS-7	[illegible]	[illegible]	N/A	22-Jan-98
OD-204	2/19/98	Polychlor.Dibenzo-p-Dioxins-Dibenzofurans-1613B Modified-MS-76	[illegible]	[illegible]	N/A	22-Jan-98
OD-205	10/7/97	Polychlor.Dibenzo-p-Dioxins-Dibenzofurans by HR/GC/MS-8290-MS-	[illegible]	[illegible]	N/A	22-Oct-97
OD-206	4/20/98	Deter.of Polychlorinated Biphenyls by Draft Method 1668	[illegible]	[illegible]	N/A	20-Apr-98
RDI RD-141	4/2/97	Radiation Safety Training	[illegible]	[illegible]	YES	10-Sep-97

ATTACHMENT 9

Supervisory Approval Memo

Memorandum

To: Employee Training File

From: [*supervisors name*]

Date:

Re: [*employees name*]

As per SWL-GA-104 Revision 5.0 (01/18/00) Employees with employment prior to June 1, 1997 are "deemed" trained to perform their duties by way of their previous work experience and this memo.

This memo officially designates SWLO employee____(*Name*)_____(*Position*)___ trained to perform the duties of his/her position by way of previous work experience either on the job here at Southwest Laboratory of Oklahoma, Inc./American Analytical & Technical Services, Inc. or from previous job experience.

_____(Supervisor Signature)____

SOUTHWEST LABORATORY OF OKLAHOMA, INC. *and*
AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.

TM

Standard Operating Procedure

Archival and Retrieval of Sample Documentation

Document No.: SWL-GA-106

Rev No. / Date: Rev. 2.1 — 05/22/00

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APPROVALS	DATES
Chuck Hoover Procedure Prepared By	5/30/00 Date
Steve L. Mawhinney Laboratory Manager or Director	06/01/00 Date
Bryan Hirsch Laboratory Safety Officer	6/01/2000 Date
Chuck Hoover Laboratory QA/QC Officer	5/30/00 Date

(Effective Date is 10 calendar days after the last signature above - QA/QC Officer)

Document Status

[ ] Controlled DC No. _____ Issued to: _____ Date: _____ QA Initials: _____

[✓] Official Copy OC No.: 2431 Issued to: Linda Karonowich Date: 11/14/00 QA Initials: _____

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ARCHIVAL AND RETRIEVAL OF SAMPLE DOCUMENTATION

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1.0 PURPOSE AND APPLICATION

1.1 The purpose of this document is to set guidelines and procedures for the archival and retrieval of hard copy raw data and supporting documentation generated during sample analysis. All procedures contained herein shall be consistent with the policy concerning long term storage set forth in Southwest Laboratory of Oklahoma's (SWL's) *"Preparation, Review, Revision, and Control of Procedural Documents"*, reference document I.D. SWL-GA-101.

1.2 The procedures described herein apply to all analytical testing sections within Southwest Laboratory of Oklahoma, Inc. (SWL). These sections currently include but are not limited to, GC/MS Semivolatiles, GC/MS Volatiles, GC Pesticide/PCB, GC Volatiles, HR-GC/MS Dioxin/Air, Radiochemistry, and Inorganics.

1.3 This document will also apply to any department to which any portion of the above-described data may be routed (i.e.; the General Reporting Department, Invoicing, etc.).

2.0 RESPONSIBILITIES

2.1 The preliminary archival of all hard copy data described above shall be the responsibility of the appropriate Program Managers or Section Supervisors. Subject data will be maintained within each department until such time as it is removed offsite for long term storage.

2.2 Each Program Manager or Section Supervisor may assign a designee within their department to execute the routine tasks of filing/storage and tracking of subject data. Accountability for this data, however, will remain with the appropriate Program Manager or Section Supervisor.

2.3 Each department's designated person shall record all filed data on the Archival Inventory Record form (see Attachment 1) prior to transferring boxes of data to warehouse for long-term storage.

2.4 The Document Control Officer (DCO) shall maintain its copies of the Archival Inventory Record mentioned in Section 2.3.

2.5 The system and responsibilities by which all laboratory logbooks and SOP's are maintained and archived is described in the laboratory's general and administrative SOPs, *"Preparation, Review, Revision, and Control of Procedural Documents, SWL-GA-101"* *"Archival and Retrieval of Sample Documentation"*, *SWL-GA-106.*

2.6 It is the responsibility of the Project Officer (PO) to communicate special client needs regarding on-site storage, archive storage, and disposal/return of data.

3.0 EQUIPMENT — N/A

4.0 PRECAUTIONS — N/A

5.0 PROCEDURES

5.1 Data Storage

This subsection describes archival and retrieval procedures practiced by each laboratory department, invoicing department, and QA Department.

5.1.1 For CLP type data packages, all departments shall follow the same procedures described below for the preliminary archival of hardcopy sample documentation.

NOTE: All Organic CLP type data packages not normally stored in each department must be routed and stored in the GC/MS Department, until it is ready to be archived in the warehouse. (See Section 5.1.2)

5.1.1.1 All raw data and corresponding forms are bound into data packages by Sample Delivery Group (SDG) along with any extra or miscellaneous data and stored in Perma-file storage boxes.

[Perma-File is a brand name. Any similar storage box may be utilized]

5.1.1.2 All boxes are identified with a unique number consisting of the box number and the first Episode/SDG No. contained within. Figure 1 is an illustration of a PermaProducts box end completed as specified.

(Note that the 2 alpha characters in the "Box Number " section, are based upon the "series" identification nomenclature stated in SWL-GA-101).

In the example below the ID number would be OP-1 24607.

1998 **1998**

Box Number	From	To	Destroy Date
OP – 1	24607	39111	

Contents

Client Name

ψ ψ ψ ψ ψ **PermaProducts** ψ ψ
ψ ψ ψ

Figure 1 Laboratory Department Data Storage Box Identification

OR

1998 = The year(s) data begins and ends (which shall be labeled just above the "Box Number" and "Destroy Date"

OP = Organic or Inorganic plus fraction (SERIES ID – OP; OV; IN; ETC.)

ORG = For SDG's with multiple organic fractions

1 = Box number for the Client for the whole year (i.e. OP-1 for first box, OP-2 for second, etc.)

24607 = First episode of first SDG in box
39111 = Last episode of last SDG in box

NOTE: QA/QC Department will designate a "Destroy Date"

Contents: Private Client Name; SWLO Affiliate Name (i.e. AATS); Alpha character to store miscellaneous or smaller client data (i.e. "Misc. A"; "Misc. A-B", or whatever grouping works best); or data used for multiple clients for QA-type work (i.e. "ICAL EPA", "ICAL Private", etc.)

5.1.1.3 Once boxes are full, the final SDG number shall be recorded on the box (as shown in Figure 1), and the Archival Inventory Record shall be completed and kept in a binder in the relevant department. This will be the "Original Inventory Date". Every column must be completed; however, the description column may or may not be used, per the discretion of the person performing the inventory.

NOTE: Organic hard copy data is archived in the Semivolatile Data Room.
Inorganics' onsite archives are located in its data room.
Radiochemistry's archives are located in its instrumentation room.

Boxes are archived onsite and transferred to the laboratory's offsite secured warehouse on an "as needed" basis or at a minimum of annually (see Subsection 5.2 for a full description of the offsite archival facility). *Exceptions to this will be coordinated between the PO and the Program Manager and/or his/her designee.*

Prior to transferring boxes to the warehouse, each department designee shall compare the Archival Inventory Record to the contents of the box. If the contents match the inventory list, the box is ready to be transferred. If the contents do not match the list (i.e. a data package or an episode file is missing), the missing data must be located and inserted into the box, before it can be transferred. This shall be the "Inventory Verification Date".

NOTE: It is the responsibility of the department supervisor or designee to vouch for the contents of the boxes.

Once the inventory verification is made, two copies of this completed form are made. The original will stay with the department, one copy will be filed in the very front of the box, and the third copy will be submitted to the QA/QC Designee who will keep all copies in a binder for each department. See Section 5.2 through 5.5 for following steps.

5.1.2 All Organic CLP type data packages not normally stored in each department must be routed and stored in the GC/MS Department, until it is ready to be archived in the warehouse.

See the procedures described below for the preliminary archival of hardcopy sample documentation.

5.1.2.1 All raw data and corresponding forms are bound into data packages by Sample Delivery Group (SDG) along with any extra or miscellaneous data and stored in Perma-file storage boxes.

[Perma-File is a brand name. Any similar storage box may be utilized]

5.1.2.2 All boxes are identified with a unique number consisting of the box number. Figure 2 is an illustration of a Perma Products box end completed as specified.

(Note that the 2 alpha characters in the "Box Number " section, are based upon the "series" identification nomenclature stated in SWL-GA-101).

In the example below the ID number would be OP-1 24607.

1998 **1998**

Box Number OP – 1	From	To	Destroy Date
Contents Date Filed: 6/99 Client Name: SDG# Client Name: SDG# Client Name: SDG#			
ψ ψ ψ ψ ψ **PermaProducts** ψ ψ ψ ψ ψ			

OR

Figure 2 Laboratory Department Data Storage Box Identification

1998 = The year(s) data begins and ends (which shall be labeled just above the "Box Number" and "Destroy Date"
OP = Organic or Inorganic plus fraction (SERIES ID – OP; OV; IN; ETC.)
ORG = For SDG's with multiple organic fractions
1 = Box number for the Client for the whole year (i.e. OP-1 for first box, OP-2 for second, etc.)

NOTE: QA/QC Department will designate a "Destroy Date"

Contents: Private Client Name; SWLO Affiliate Name (i.e. AATS); Alpha character to store miscellaneous or smaller client data (i.e. "Misc. A"; "Misc. A-B", or whatever grouping works best); or data used for multiple clients for QA-type work (i.e. "ICAL EPA". "ICAL Private", etc.)

5.1.2.3 Once boxes are full. the Archival Inventory Record shall be completed and kept in a binder in the relevant department. This will be the "Original Inventory Date". Every column must be completed; however, the description column may or may not be used, per the discretion of the person performing the inventory.

NOTE: Organic hard copy data is archived in the Semivolatile Data Room. Inorganics' onsite archives are located in its data room. Radiochemistry's archives are located in its instrumentation room.

Boxes are archived onsite and transferred to the laboratory's offsite secured warehouse on an "as needed" basis or at a minimum of annually (see Subsection 5.2 for a full description of the offsite archival facility). *Exceptions to this will be coordinated between the PO and the Program Manager and/or his/her designee.*

Prior to transferring boxes to the warehouse, each department designee shall compare the Archival Inventory Record to the contents of the box. If the contents match the inventory list, the box is ready to be transferred. If the contents do not match the list (i.e. a data package or an episode file is missing), the missing data must be located and inserted into the box, before it can be transferred. This shall be the "Inventory Verification Date".

NOTE: It is the responsibility of the department supervisor or designee to vouch for the contents of the boxes.

Once the inventory verification is made, two copies of this completed form are made. The original will stay with the department, one copy will be filed in the very front of the box, and the third copy will be submitted to the QA/QC Designee who will keep all copies in a binder for each department. See Section 5.2 through 5.5 for following steps.

5.1.3 For non-CLP type reports (i.e.; standard tabular) all hardcopy data is routed to SWL's General Reporting Department. Once reported, the reports are copied (at least one copy is made for the laboratory) and transferred to the Invoicing Department.

5.1.3.1 Once invoicing is complete, the Invoicing Clerk (IC) files all hardcopy data. They are maintained in lateral file cabinets located in the invoicing department. Rotation of data from lateral files to archival storage boxes is at the discretion of the Invoicing Clerk. Boxes are to be maintained in the Invoicing department area until they are transferred to the warehouse.

5.1.3.2 Boxes are identified differently for non-CLP reports (see Figure 3).

Episode
37405 to 37844

1998 1998

Box Number	From	To	Destroy Date
INV – 1	Mar. 99	Apr. 99	

Contents

Client Name

ψ ψ ψ ψ ψ PermaProducts ψ ψ
ψ ψ ψ

Figure 3 Reporting Dept. and Invoicing Storage

Episode = The range of SDG numbers from the smallest to the largest
1998 = The year(s) data begins and ends (which shall be labeled just above the "Box Number" and "Destroy Date"
INV = The Invoicing Department's abbreviation.
1 = Box number for the Client for the whole year (i.e. INV-1 for first box, INV-2 for second, etc.)
Mar. 99 = Month and year taken from first episode/SDG in the box.
Apr. 99 = Month and year taken from last episode/SDG in the box.

NOTE: QA/QC Department will designate a "Destroy Date"

Contents: Private Client Name; SWLO Affiliate Name (i.e. AATS); Alpha character to store miscellaneous or smaller client data (i.e. "Misc. A"; "Misc. A-C", etc.)

5.1.3.3 On an "as needed" basis, current year boxes will be transferred from the Invoicing department to one of two storage rooms located within the General Reporting area. This onsite move will remain the responsibility of the Invoicing Clerk.

5.1.3.4 Overflow from the current year's reports, as well as reports from the previous year, are kept onsite in the storage rooms described in 5.1.2.3. For example, all of 1996 General Reports will reside in the storage rooms as 1997 is accumulating. In January of 1998 all the 1996 boxes will be transferred to our offsite facility for long term storage; 1997 will remain in the storage rooms within the General Reporting area as 1998 begins to accumulate.

NOTE: All boxes will be kept in the Laboratory's offsite warehouse in archive status for 5 years, at which time they will be transported to a predetermined paper recycling site.

5.1.3.5 *Exceptions to the following will be coordinated between the PO and the IC & DCO.* Prior to transferring boxes from the Invoicing Clerks jurisdiction to the offsite storage warehouse, the Invoicing Clerk must complete the "Archival Inventory Record" (see Section 6.0 Records, for instructions). Upon completion, two copies will be made: one copy is filed in the front of the box; one copy is submitted to the QA/QC Designee; and the original will remain with the Invoicing Clerk.

Before boxes can be transferred to the warehouse, the Invoicing Clerk shall compare the Archival Inventory Record to the contents of the box. If the contents match the inventory list, then the box is ready to be transferred. If the contents do not match the list (i.e. a data package or an episode file is missing), then the missing data must be located and inserted into the box, before it can be transferred.

5.2 Description of Offsite Storage Facility.

The offsite storage or warehouse building is located several miles from our main laboratory facility at 3101 West Albany, Broken Arrow, Oklahoma

With offices located in the front and warehouse space located in the back Southwest Laboratory's storage facility covers approximately 18,143 sq. ft.. Of the total area, 14,584 sq. ft. is dedicated warehouse space.

The entire building is climate controlled. Other features include a sprinkler system for fire protection, recessed loading dock, and a security system.

Access to the building is tightly controlled. Keys are maintained by the Laboratory Director, and the Laboratory Operations Manager.

5.3 Prior to transferring archive data from onsite storage to the warehouse, an assigned employee must first identify and remove all boxes 5 years old or more. These boxes are to be taken to the predetermined recycling facility for disposal. *Exceptions to this will be coordinated between the PO and the DCO as detailed in the above sections: 5.1.1.3; 5.1.2.3; 5.1.3.3 & 5.1.3.5.*

NOTE: All data 5 years old or more must be properly destroyed as per this section. No data shall be thrown in the trash or otherwise compromised. Client confidentiality is always upheld.

Once space is available, the archived data from the lab may be stacked in the appropriate shelves. (See Section 5.5)

5.3 The process described in Section 5.3 shall be performed on an annual basis (ideally in the Fall), at a time to be determined by the QA/QC Officer and QA DC-Designee.

5.4 Any employee assigned the responsibility of transporting and storing any laboratory data must be able to identify which boxes are to be stored in the Laboratory Data Storage area and which will be stored in the Invoicing Data Storage area. To assure easy identification. each box must be labeled exactly as described in Sections 5.1.1.2 & 5.1.2.2.

The data storage area of the warehouse is divided into two clearly labeled sections: Laboratory Data Storage and Invoicing Data Storage. The archived data boxes are stored on large steel-framed shelves.

5.4.1 All laboratory data boxes will be stored in the Laboratory Data Storage section of the warehouse.

These boxes will be grouped by year from oldest to most recent and then stacked in alphabetical order by client name, and subdivided by department series.

i.e. 1995
→ Misc. "A"
AATS
→ IN-1, IN-2, OG-1, OG-2, OP-1, OP-2, OP-3, etc.

5.4.2 All invoicing data boxes will be stored in the Invoicing Data Storage section of the warehouse.

These boxes will be grouped by year from oldest to most recent and then stacked in alphabetical → order by client name.

i.e. 1995
→ Misc. "A"
AATS
INV-1, INV-2, etc.

5.5 Data Retrieval

Retrieval of Data from long-term storage will be achieved through a request to the QA/QC Officer, who will notify the Operations Manager.

5.5.1 The individual requesting access to archived data boxes must complete the Data Retrieval Form (see Attachment 2), and submit it to the QA/QC Officer for approval. If the request is approved, the QA/QC Officer will notify the Operations Manager and then sign the "Data Retrieval Form" and submit it to the QA DC-Designee. The QA DC-Designee must maintain the form and have the requester sign it again once all boxes, files, data, etc. are returned. If the request is denied, the QA/QC Officer must advise the requester.

NOTE: All boxes (and its contents) must be returned in a reasonable length of time. If they are needed for a period longer than 2 weeks, this must be noted on the form. The QA DC-Designee will e-mail reminders to the requester on a weekly basis until all data is returned.

5.5.2 The DCO will keep all the Data Retrieval Forms in a binder.

6.0 RECORDS

6.1 An Archival Inventory Record (see Attachment 1) shall be completed for any Perma File Box containing documentation generated during sample analysis. All departments shall maintain the appropriate Archival Inventory Record forms, a copy of which must be submitted to the QA Department upon request for transfer [of subject boxes] offsite for long term archival.

Responsibility for the initiation and completion of Archival Inventory Record (see Attachment 1) may be delegated by the individual Program Managers/Section Supervisors.

6.2 A Data Retrieval Form (see Attachment 2) must be completed by the individual requesting retrieval of archived data. See Section 5.6 for details.

7.0 REFERENCES

7.1 Standard Operating Procedure, document I.D. - SWL-GA-101, "*Preparation, Review, Revision, and Control of Procedural Documents*"

8.0 DEFINITIONS — N/A

9.0 ATTACHMENTS

Attachment 1: Archival Inventory Record

Attachment 2: Data Retrieval Form

ATTACHMENT 1
Archival Inventory Record
(Example Only)

Archival Inventory Record, SWL-GA-106

GENERAL ADMINISTRATIVE

SOP SWL-GA-106, REV. # 2.0 FILE ID: GA106a1.doc .

Department: _____

Program Mgr./Section Supervisor: _____

_____ *Original Inventory Performed by*

_____ *Original Inventory Date*

_____ *Inventory Verification Performed by*

_____ *Inventory Verification Date*

Year	Box ID	SDG	Listing of Episode	Brief Description of Contents

SOUTHWEST LABORATORY OF OKLAHOMA, INC.
1700 West Albany • Broken Arrow, OK 74012 • Office (918) 251-2858 • Fax (918) 251-2599

ATTACHMENT 2
Data Retrieval Form
(Example Only)

Data Retrieval Form, SWL-GA-106

GENERAL ADMINISTRATIVE

SOP SWL-GA-106, REV. # 2.0 FILE ID: GA106a2.doc

Department: _____

Program Mgr./Section Supervisor: _____

_____ *Retrieval Requester* _____ *Date*

_____ *QA/QC Officer* _____ *Approval Date*

_____ *Retrieval Requester return signature* _____ *Return Date*

Year	Box ID	SDG	Listing of Episode	Brief Description of Contents Needed & Reason for Retrieval
1997	OP-1 29705	27705	27705	(1) SDG only, 3 volumes — Pesticide data needed
				for Revision or Corrective Action.
1998	OP-3 47221	47221	47221	(3) in SDG, — Pesticide data needed for Revision
			47229	or Corrective Action
			47230	

Additional Comments: *(Please use the space provided to explain your need to keep the archived data for longer than two weeks.)*

_____

_____

_____

_____

SOUTHWEST LABORATORY OF OKLAHOMA, INC.
1700 West Albany • Broken Arrow, OK 74012 • Office (918) 251-2858 • Fax (918) 251-2599

SOUTHWEST LABORATORY OF OKLAHOMA, INC.

TM

Standard Operating Procedure

Procurement of Materials and Equipment

Document No.: SWL-GA-107

Rev No. / Date: Rev. 1.2 — 10/16/98

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APPROVALS	DATES
Chuck Howes Procedure Prepared By	10/16/98 Date
Steve L. Marsh Laboratory Manager or Director	10/23/98 Date
Chuck Howes Laboratory Safety Officer	10/16/98 Date
Chuck Howes Laboratory QA/QC Officer	10/16/98 Date

(Effective Date is 5 calendar days after the last signature above - QA/QC Officer)

Document Status

[ ] Controlled	DC No. _____	Issued to: _____	Date: _____	QA: _____
[✓] Official Copy	OC No.: 2582	Issued to: Tom Johnston	Date: 05/05/02	QA: [illegible]

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PROCUREMENT OF MATERIALS AND EQUIPMENT

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1.0 PURPOSE AND APPLICATION

1.1 The purpose of this document is to establish a system to assure that products and services purchased or contracted for by Southwest Laboratory of Oklahoma, Inc. (SWL) shall meet at least the minimum standards required.

1.2 This procedure is applicable to all materials, equipment, or services procured by SWL which could affect the quality and consistency of the work performed by the laboratory. Detailed instructions and guidelines for requesting materials, equipment, or services; the purchase approval process; and the receipt inspection and acceptance process are outlined herein. Along with a detailed description of the system which shall ensure that reagents, and solvents used during analysis are of appropriate purity.

2.0 RESPONSIBILITIES

2.1 The Laboratory Director or Laboratory Manager are responsible for the approval of all Purchase Requisitions.

2.2 The Laboratory QA/QC Officer is responsible for the incorporation of quality assurance program requirements appropriate to the procurement of materials which could have an impact on the quality of work performed at Southwest Laboratory of Oklahoma (i.e.: reagents, solvents, standards, and gases).

2.3 The Purchasing Officer is responsible for the control of requisition forms, the subsequent purchase orders issued, and the reconciliation of orders to actual materials, equipment, or services received.

2.4 The Requester (any SWL employee who submits a procurement request) is responsible for the inspection of the package/materials when received.

2.5 All Southwest Laboratory employees who make procurement requests and/or receive items and all supplying vendors are responsible for following the applicable sections of this procedure.

3.0 EQUIPMENT. None.

4.0 PRECAUTIONS. Chemicals and instrumentation should be regarded as potentially hazardous, and therefore, should be handled with caution to ensure safety and prevent contamination.

5.0 PROCEDURES

5.1 Procurement Request

5.1.1 The Requester of materials shall complete a "*Purchase Requisition Form*" (see Attachment 1) based on the quality of materials specified in its applicable SOP. Proper completion of the form shall include catalog numbers and a description of the material requested. Descriptions will include the grade of

chemical. the tolerances of the glassware and measurement devices and/or the type of supplies required.

5.1.2 The Program Manager/Section Supervisor is required to review and initial the Purchase Requisition to ensure that proper quality requirements are met.

5.1.3 The completed purchase request shall be submitted to the Laboratory Director/Manager for approval of the purchase.

5.1.4 The approved purchase requisition shall be forwarded to the Purchasing Officer who will place the order and keep the purchase requisition on file until the order is received. If a product is out of stock the Purchasing Officer will notify the Requester so a product of equal quality can be ordered.

5.1.5 Prior to the purchase of Methylene Chloride Lots, the vendor is required to supply a bottle from the subject Lot. The solvent will be checked for any impurities that may affect sample analysis. If an impurity is found the lot will not be purchased and another bottle will be supplied from a different lot. Once a clean lot is found the supplier will hold that lot until it is diminished. This data is maintained by the Extraction's Supervisor.

5.1.6 Factors considered in making purchase requisitions shall include: 1) the identity, purity, shelf-life, and source; 2) tests to be conducted for quality and purity; 3) storage and handling procedures; and 4) the replacement dates.

5.1.8 Sources of supplied equipment materials or services must be selected with care. Only suppliers that have a history of providing identical or similar products or services which perform satisfactorily in actual use will be used. The suppliers history shall reflect current capability. If a supplier is being considered whose history is not known, a sample of the product will be provided for quality testing. If the product passes quality inspection then the new supplier can be used.

5.2 Subcontracting Analytical and/or Technical Services

5.2.1 In the event subcontracted analytical services are required, the procurement process shall be governed by SWL's Small Business Subcontracting Plan. The plan includes an historical list of small businesses which have performed to specifications in the past, and the utilization of a source provided by the Small Business Administration (SBA). The current plan is maintained by the laboratory's Manager of the Contracts/Proposals Department.

5.2.2 It is the responsibility of the Manager of Contracts/Proposals to write a solicitation which shall include all requirements provided by the Prime Contractor for whom the subcontracted work will be performed. The requirements of the solicitation (or Request for Proposal - RFP) may include, but are not limited to the following:

5.2.2.1 Any required State and/or Federal Agency certifications, validations, and/or the successful completion of Performance Evaluation samples, and on-site audits.

5.2.2.2 The applicable scope of work and quality assurance objectives from the Prime's requirements of Southwest Laboratory of Oklahoma. In all cases, Southwest Laboratory must confirm that any subcontracted analytical/technical firm shall meet or exceed all requirements as specified by its Prime Contractor.

5.3 Receipt of ordered materials.

5.3.1 Upon receipt of ordered materials, the Requester shall perform the following:

5.3.1.1 Inspect for damages or breakage.

5.3.1.2 Check packing slip versus the item(s) received to verify that they match and are as ordered.

5.3.1.3 Sign and date the packing slip indicating approval to the Purchasing Officer.

5.3.1.4 Sign and date all laboratory reagents.

5.3.2 The Requester is responsible for verifying upon receipt that the materials delivered do in fact meet specifications; this check may be based upon visual inspection of materials and specifications; or may involve actual tests performed upon them to establish their acceptability.

5.3.3 Reagents and chemicals are tested as reagent blanks to show that the levels of contamination are below levels of concern for the methods they will be used with. The lot numbers for reagents and chemicals used in the preparation of samples for analysis will be noted in either the preparation logbook or the Organic Extraction Solvent Reagent Log (attachment 3).

5.3.4 New measurement devices are calibrated and checked against reference sources using ASTM or other established tests.

5.3.5 Any items received which could have an impact on the quality of work performed (i.e.: reagents, solvents, standards, and gases, or analytical/technical services) and do not conform to specifications will be returned to the vendor for replacement. However, if urgent, then attempts to upgrade the materials to meet specifications shall be made through any available means. Discrepancies shall be entered on a Nonconformance Form (see SWL-GA-105, *"Corrective Actions"*) and submitted initially to the Purchasing Officer. Once the Purchasing Officer has either returned or upgraded the materials, he/she shall forward *"Procurement Nonconformance Forms"* (see Attachment 2) to the QA Department for filing and tracking by the QA/QC Officer.

5.3.6 Laboratory reagents obtained from approved commercial vendors shall meet ACS Standards. SWL shall assure reagents received are labeled showing contents, date opened and by whom, and where appropriate, expiration date.

6.0 RECORDS

6.1 Purchase Requisition Forms shall be maintained by the Purchasing Officer.

6.2 Nonconformance Reports shall be filed in the quality assurance file and tracked by the Quality Assurance Officer.

7.0 REFERENCES. None

8.0 ATTACHMENTS

8.1 Attachment 1 – Reference 5.1.1 "Purchase Requisition Form"

8.2 Attachment 2 – Reference 5.3.5 "Procurement Nonconformance Form"

8.3 Attachment 3 – Reference 5.3.3 "Organic Extraction Solvent Reagent Log"

9.0 DEFINITIONS

9.1 Terminology

Lots ..... Any items, materials, or goods produced in controlled batches for traceability.

Shelve Life ..... The expiration date either assigned by manufacturer or by lab personnel as produced/received.

Packing Slip ..... Documentation from the Vendor and included with the shipped package itemizing all items included according to the Purchase Order issued by the laboratory, including any back orders (not available at the time of shipment – but should include the future ship date).

9.2 Acronyms

ASTM ..... *"American Society for Testing and Materials"*, Reference book.

ACS ..... *"American Chemical Society"*, organization

PURCHASE REQUISITION

Procurement Form (107-ATT1.doc) Rev. 1.0-05/19/97 Form ID: GA-107-PURCH-F

BROKEN ARROW LOCATION

PURCHASING DEPARTMENT USE ONLY: P.O.# | P.O. Date

VENDOR *only one vendor per requisition*

Suggested Vendor: _____ Phone: _____

Address: _____ Fax: _____

_____ Contact: _____

ORDER INFORMATION / COST

QUANTITY	CATALOG NUMBER	DESCRIPTION	QTY. PER CS OR PK	ESTIMATED AMOUNT UNIT PRICE	ESTIMATED AMOUNT TOTAL	ACTUAL AMOUNT UNIT PRICE	ACTUAL AMOUNT TOTAL	EXPECTED DELIVERY DATE
				TOTAL:		TOTAL:		

CHECK ONE: ☐ Consumable Supplies ☐ Replacement Parts ☐ New Equipment

SHIP VIA: ☐ Standard Delivery ☐ Next Day Air ☐ 2nd Day Air

REASON FOR PURCHASE: _____

SPECIAL INSTRUCTIONS: _____

_____ TERMS: _____

CHARGE TO DEPT. (if different than requesting dept.) _____

Requested by: _____ Date: _____

Department: _____

Approved by Dept. Mgr: _____ Date: _____

All orders require approval by Rich Ronan, Rob Harris, or Jack Wright:

Approval: _____ Date: _____

SOUTHWEST LABORATORY OF OKLAHOMA, INC. • 1700 W. ALBANY • BROKEN ARROW, OKLAHOMA 74012 • OFFICE (918) 251-2858 • FAX (918) 251-2599 [AC028-0593-01]

ATTACHMENT 1 – Purchase Requisition Form

Typist: LLR

NONCONFORMANCE REPORT, SWL-GA-107

Procurement Form (107-ATT2.pm) Rev.1.0 - 05/19/97 Form ID: GA-107-NONCON-F

Documentation of the Receipt of Nonconforming Materials Effecting Quality of Work.

FROM:
PURCHASING DEPARTMENT

TO:
QA/QC OFFICER
QA DEPARTMENT

Date: _____ Date Received: _____

From: _____ PO Number: _____

Dept: _____ PO Date: _____

ITEMS TO BE RETURNED **VENDOR:**

QUANTITY	CATALOG NUMBER	DESCRIPTION

REASON FOR RETURN

_____

_____

_____

Approved by: _____ (Department Manager)

TO BE COMPLETED BY THE PURCHASING DEPARTMENT

Date: _____ Shipping Address: _____

Invoice #: _____ _____

Contact: _____ _____

Return Authorization: _____ _____

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[AC029-0593-01]

ATTACHMENT 2 – Nonconformance Report Form

ORGANIC EXTRACTION DEPARTMENT

ORGANIC EXTRACTION SOLVENT REAGENT LOG

Ref: SWL-GA-107 Rev. 1.0
Book ID # GA-107-ESL-1
F:\COMMON\ORGANICS\OEXTSRL.DOC

Reviewed By_____

PAGE #_____ OF BOOK # 1

	SOLVENTS / REAGENTS	LOT NUMBER	DATE LOT STARTED	DATE LOT ENDED	LOT CONTINUED NEXT PAGE YES / NO	
	METHYLENE CHLORIDE					
	ACETONE					
	HEXANE					
	CYCLOHEXANE					
	ETHER					
	ACETONITRILE					
	BENZENE					
	TOLUENE					
	CHLOROBUTANE					
	SUDIUM SULFATE					
	SODIUM CHLORIDE					
	SODIUM HYDROXIDE					
	POTASSIUM CHLORIDE					
	SILICA GEL					
	ALUMINA					
	SULFURIC ACID					
	HYDROCHLORIC ACID					

ATTACHMENT 3 – Organic Extraction Solvent Reagent Log

SOUTHWEST LABORATORY OF OKLAHOMA, INC. *and*
AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.

TM

Standard Operating Procedure

Performance and Systems Audits

Document No.: SWL-GA-108

Rev No. / Date: Rev. 5.0 — 07/21/00

OFFICIAL COPY RED STAMP INDICATES ORIGINAL

APPROVALS	DATES
Chuck Homer Procedure Prepared By	9/21/00 Date
Steve L. Markham Laboratory Manager or Director	09/29/00 Date
Bryan Tucker Laboratory Safety Officer	9/22/2000 Date
Chuck Homer Laboratory QA/QC Officer	9/21/00 Date

(Effective Date for is 5 calendar days after the last signature above - QA/QC Officer)

Document Status

[ ] Controlled DC No. _____ Issued to: _____ Date: _____ Initials: _____

[✓] Official Copy OC No.: 2431 Issued to: Linda Karsonovich Date: 11/14/00 Initials: LK

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PERFORMANCE AND SYSTEMS AUDITS

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1.0 PURPOSE AND APPLICATION

1.1 This document gives instruction in preparing, conducting and reporting of Laboratory Performance and System Audits for Southwest Laboratory.

1.2 This procedure is applicable to all procedures, process, and analysis in the Laboratory. This procedure is not to be limited by the items listed in the checklists.

2.0 RESPONSIBILITIES

2.1 The individual program managers are responsible to correct the deficiencies found by the audits.

2.2 The Laboratory Manager is responsible to assure that the laboratory is ready for an audit at any time and assist in the scheduling and any preparation needed.

2.3 The Laboratory Quality Assurance Officer is responsible for scheduling, coordinating and conducting quarterly system audits, preparing blind performance audits, and providing the results of the audits. The Laboratory Quality Assurance Officer is also responsible for reporting the results of the audit to the Laboratory Director with a copy to the Laboratory Manager.

2.4 The Program Managers are responsible for providing the personnel who are needed to provide the information required during system audits.

3.0 EQUIPMENT REQUIRED — N/A

4.0 PRECAUTIONS — N/A

5.0 PROCEDURE

5.1 Performance Audits — Procedures used to assess the effectiveness of the quality control system are as follows:

5.1.1 Internal Performance Audits — These are accomplished by the laboratory through the introduction of blind Performance Evaluation samples.

5.1.2 External Performance Audits — These are accomplished by the laboratory through inter-laboratory checks such as:

5.1.2.1 Participation in various state laboratory evaluations programs.

5.1.2.2 Participation in a NIST approved PT provider WS, WP, and solid or soil studies. These P.E. studies are to be analyzed as a typical samples (no multiple analyses). An attestation statement regarding the analyses of these P.E.'s will be signed by the Laboratory Director or QAO as provided by the vendor.

5.1.2.3 Analysis of split samples, and a comparison of the results with the other laboratory.

5.1.2.4 Participation in the US EPA CLP program. The CLP program includes quarterly blinds as well as regional case specific blinds.

5.1.2.5 Participation in the US Army Corps of engineers DERA (Defense Environmental Restoration Agency) certification program.

5.1.2.6 Participation in DOE's – INEL MAPEP Performance Evaluation Program.

5.2 System/Technical Audits

5.2.1 Coordinate the time for a system/technical audit with the Laboratory Manager and Program Managers.

5.2.2 Schedule the areas and procedures so that a complete audit of all the areas and procedures will be conducted during a calendar year. Any areas that have discrepancies are to be checked during the next audit time to assure that those discrepancies have been corrected. (See Attachments 3 & 4 — Audit schedules.)

5.2.3 Schedule and list the procedures or functions that shall be audited for the particular audit. Provide a copy of the schedule to the Laboratory Manager so that proper use of technicians' time can be maintained.

Have a meeting, some days in advance, with the managers whose areas shall be audited to indicate what shall be expected for this particular audit. The personnel may then prepare and have the particular audit. The personnel may then prepare and have the necessary information nearby for a more expeditious audit.

5.2.4 For technical audits, review the method versus the SOP. Make sure any deviations are noted in the Scope and Application of the procedure. For systems audits, use the SOP as the checklist.

5.2.5 Using the checklist, perform an audit indicating information on the checklist to conditions found. (See Attachment 1 or system SOP.)

5.2.5.1 If an item of finding, observation, or comment is discovered make note on the checklist page to document the information.

5.2.5.2 If a condition that can be considered adversely affecting quality is discovered, a corrective action form (SWL-GA-105) is to be initiated by the responsible manager. Depending on the severity of the situation immediate action may be taken to prevent further processing of data until corrected.

5.2.5.3 If a procedural non-conformance is found, a non-conformance report (SWL-GA-105) is initiated.

5.2.5.4 An Internal Audit Report is produced detailing the findings, corrective actions (CARs) and/or any non-conformance (NCRs). The

reports are sent to the Department Heads for investigation/follow-ups/corrections.

5.2.6 The Program Managers are to respond to the audit report with corrective action to be taken. If a method deviation is noted, this deviation will be addressed in the SOPs Scope and Application. These deviations will be forwarded to reporting to include in case narratives when applicable.

5.2.7 A follow-up inspection for the items of the response shall be conducted shortly after the corrective action completion with the report to the addresses of the original audit report. Also as in item 5.2, any areas that have discrepancies are to be checked during the next audit time to assure that those discrepancies have been corrected.

6.0 RECORDS

6.1 Results from Blind Performance Evaluating Samples are to be maintained by the QA Department

6.2 Audit checklists are to be completed by the Laboratory Quality Assurance Officer and maintained in the QA files.

6.3 Internal Audit Reports are to be prepared by the Laboratory Quality Assurance Officer and maintained in the QA files.

6.4 The response(s) by the Program Managers is/are to be maintained in the QA files.

7.0 REFERENCES

7.1 Applicable Work Area Standard Operating Procedures

7.2 Methodology used in establishing individual Standard Operating Procedures.

8.0 DEFINITIONS

Finding:..... A procedural nonconformance indication that a critical requirement was not being met or had not been effectively implemented.

Observation:..... A potential problem, which if left uncorrected, could result in a condition adverse to quality.

Comment:..... A laboratory condition, positive, or negative noted during the audit.

NIST:..... National Institute of Standards and Technology.

PT Provider:..... Proficiency Testing Provider

WS: ..... Water Supply

WP: ..... Water Pollution

QAO:..... Quality Assurance Officer

DOE: ..... Department of Energy

INEL: ..... Idaho National Engineering and Environmental Laboratory

MAPEP: ..... Mixed Analyte Performance Evaluation Program

9.0 ATTACHMENTS

- Attachment 1 — Chemistry Test Methods Checklist
- Attachment 2 — Internal Audit Report
- Attachment 3 — Technical Audit Schedules
- Attachment 4 — System Audit Schedules

ATTACHMENT 1
Chemistry Test Methods Checklist

CHEMISTRY TEST METHODS

Note: Make enough copies of Pages 5-13 to assess each test method in use at the laboratory, one method at a time. In addition, copy Page 14 to assess each chromatography method & Page 15 for each PBMS test method

TEST METHOD EVALUATED: _____

___	5.10.1.1	Does the laboratory have standard operating procedures for all Chemistry test methods Note: These documents may be equipment manuals provided by the manufacturer
___	5.10.1.1(c)	Are copies of these standard operating procedures accessible to all personnel
___	5.10.1.1(e)	Does each standard operating procedure indicate the effective date of the document, revision number, & signature of the approving authority
___	5.10.1.1(d) 5.10.1.2(a)	Does the laboratory have an in-house methods manual for each accredited analyte or method Note: This manual may consist of copies of published or referenced test methods
___	5.10.1.2(b)	Does the laboratory clearly indicate in its methods manual any modifications made to the referenced test method and describe any changes or clarifications where the referenced test method is ambiguous or provides insufficient detail

Does each test method in the in-house methods manual include or reference, where applicable:

___	5.10.1.2(b)(1)	Identification of the test method
___	5.10.1.2(b)(2)	Applicable matrix or matrices
___	5.10.1.2(b)(3)	Method Detection Limit
___	5.10.1.2(b)(4)	Scope & application, including components to be analyzed
___	5.10.1.2(b)(5)	Summary of the test method
___	5.10.1.2(b)(6)	Definitions
___	5.10.1.2(b)(7)	Interferences
___	5.10.1.2(b)(8)	Safety
___	5.10.1.2(b)(9)	Equipment & supplies
___	5.10.1.2(b)(10)	Reagents & standards
___	5.10.1.2(b)(11)	Sample collection, preservation, shipment, & storage
___	5.10.1.2(b)(12)	Quality control
___	5.10.1.2(b)(13)	Calibration & standardization
___	5.10.1.2(b)(14)	Procedure
___	5.10.1.2(b)(15)	Calculations
___	5.10.1.2(b)(16)	Method performance
___	5.10.1.2(b)(17)	Pollution prevention
___	5.10.1.2(b)(18)	Data assessment & acceptance criteria for quality control measures
___	5.10.1.2(b)(19)	Corrective actions for out-of-control data
___	5.10.1.2(b)(20)	Contingencies for handling out-of-control or unacceptable data
___	5.10.1.2(b)(21)	Waste management
___	5.10.1.2(b)(22)	References
___	5.10.1.2(b)(23)	Tables, diagrams, flowcharts, validation data

___	5.5.4(d)	Does the laboratory's Chemistry data indicate that the quality control protocols in the test methods manual are being followed
___	D.1.5	Has the laboratory documented procedures for data reduction (e.g. use of linear regression)

COMMENTS:

TEST METHOD EVALUATED: _____

___ 5.10.5(d) Do all containers of prepared reagents & standards have a unique identifier & expiration date that links these specific containers of reagents & media to their preparation records
Note: See pages 17-37 for the reagents & standards required for each test method, along with the required purities & shelf-life

___ D.1.6(a) Does the laboratory maintain records of calibration certificates, traceability to national standards of measurement, measurement results with associated uncertainties, or correlations of results for each standard source needed
Note: These Standards are from NELAC 5.9.2

___ D.1.6(b)(1) Does the laboratory use analytical reagent grade materials when the purity of reagents is not specified in the test method

___ D.1.6(b)(1) Does the laboratory not use reagents of lesser purity than those specified by the test method

___ D.1.6(b)(1) Does the laboratory document any checks it makes to verify that the purity of reagents meets the test method requirements

___ D.1.6(b)(2) Does the quality of the laboratory's reagent water meet method-specified requirements

COMMENTS:

___ 5.11.3(a)(2) Has the laboratory checked samples for proper preservation (e.g. pH, absence of free chlorine, temperature) prior to or during sample preparation or analysis
Note: See pages 38-40 for holding times, sample containers allowed, & preservation required for each analyte

COMMENTS:

TEST METHOD EVALUATED: _____

Note: The following Standards for initial instrument calibration & calibration verification are also referenced in NELAC D.1.3(b)

___	5.9.4.2.1(a)	Does the laboratory's test method SOP include or reference details of the initial instrument calibration procedures Note: This includes calculations, integrations, & associated statistics
___	5.9.4.2.1(b)	Does the laboratory retain sufficient raw data records to permit reconstruction of the initial instrument calibration Note: Examples of such data records include calibration date, test method, instrument, analysis date, each analyte name, concentration & response, & calibration curve or response factor
___	5.9.4.2.1(c)	Does the laboratory quantitate sample results only from the initial instrument calibration and not from any continuing instrument calibration verifications
___	5.9.4.2.1(d)	Does the laboratory verify all initial instrument calibrations with a standard obtained from a second source
___	5.9.4.2.1(e)	Has the laboratory established criteria for the acceptance of an initial instrument calibration Note: Examples include linear regression correlation coefficient & response factor %RSD
___	5.9.4.2.1(f)	Does the laboratory report sample results not bracketed by initial instrument calibration standards as having less certainty Note: Data qualifiers, flags, and/or explanations in the case narratives may be used to comply with this standard
___	5.9.4.2.1(f)	Is the lowest calibration standard above the detection limit for each analyte
___	5.9.4.2.1(g)	Does the laboratory perform corrective actions if the initial instrument calibration results are outside established acceptance criteria
___	5.9.4.2.1(g)	Does the laboratory not report sample data associated with unacceptable initial instrument calibrations Note: NELAC Standards 5.9.4.2.1(f) & (g) may need to be assessed in conjunction with the Quality Systems data audit
___	5.9.4.2.1(h)	Do the calibration standards include concentrations at or below regulatory limits or decision levels Note: This Standard applies if such regulatory concentrations are known by the laboratory Note: This Standard does not apply if these regulatory concentrations are below the laboratory's demonstrated detection limits; however, NELAC Standard 5.1(b) may apply
___	5.9.4.2.1(i)	Does the laboratory have a standard operating procedure for determining the number of points for establishing the initial instrument calibration
___	5.9.4.2.1(f)	Does the laboratory use a minimum of two concentration points (plus blank) for performing an initial instrument calibration
___	5.1(b)	Does the laboratory fulfill the initial instrument calibration requirements contained in mandated test methods & regulations (see pages 41-42 for acceptance criteria & the number of standards required)

TEST METHOD EVALUATED: _____

___	5.9.4.2.2	Does the laboratory verify the validity of the initial calibration by a **continuing instrument calibration verification** with each **analytical batch**, prior to **sample analyses**, whenever an initial instrument calibration is not performed on the day of analysis
___	5.9.4.2.2(a)	Are the details of the continuing instrument calibration verification **procedure, calculations, & associated statistics** included or referenced in the test method SOP
___	5.9.4.2.2(b)	Is the continuing instrument calibration verification repeated at the **beginning & end of each analytical batch** **Note:** Only one calibration verification analysis is required per batch if an **internal standard** is used
___	5.9.4.2.2(b)	Are the concentrations of the continuing calibration standard varied within the **established calibration range**
___	5.9.4.2.2(c)	Does the laboratory retain **sufficient raw data records** to permit **reconstruction** of the continuing instrument calibration verification **Note:** Such records include test method, instrument, analysis date, name of each analyte, concentration & response, and calibration curve or response factor
___	5.9.4.2.2(d)	Has the laboratory established **criteria for the acceptance** of a continuing instrument calibration verification (e.g. relative percent difference)
___	5.9.4.2.2(e)	Does the laboratory perform **corrective actions** if the continuing instrument calibration verification results are **outside established acceptance criteria**
___	5.9.4.2.2(e)	Does the laboratory perform a **new initial instrument calibration** if the routine corrective action **procedures fail to produce a second consecutive** (immediate) calibration **verification within acceptance criteria**
___	5.9.4.2.2(e)	Under such circumstances, do additional sample analyses not occur until a **new initial instrument calibration** is established & verified **Note:** For sample data associated with an **unacceptable** calibration verification, the following test results may be reported as **qualified data:** - **Non-detects** for analytes in associated samples where the acceptance criteria for the continuing calibration verifications are **exceeded high** - Any test result for an analyte that indicates **exceedence of a maximum regulatory limit** or decision level, when the acceptance criteria for the continuing calibration verification for that analyte is **exceeded low** Any samples with test results that do not meet either of the above criteria **must be re-analyzed after a new initial instrument calibration** has been **established, evaluated, & accepted**
___	5.1(b)	Do the laboratory's continuing instrument calibration verifications meet the requirements in **mandated test methods & regulations** (see pages 43-44 for acceptance criteria)

COMMENTS: List individual analytes and specific details where the above NELAC Standards are **not being fulfilled**

TEST METHOD EVALUATED: _____

___	5.10.2.1(a) D.1.3(a)	Has the laboratory performed a satisfactory demonstration of method capability prior to the acceptance & insutution of this test method Note: These Standards are not applicable for tests with which spiking is not an option and for which Quality Control samples are not readily available (e.g. pH, odor, temperature, & dissolved oxygen)
___	C.1(a)	Is the quality control sample used for this Demonstration of Capability obtained from an outside source Note: If an outside source is not available, the laboratory may prepare this sample with stock standards that are prepared independently from those used in instrument calibration
___	C.1(b)	Are the analytes diluted in a volume of clean matrix sufficient to prepare 4 aliquots at the required method sample volume to a concentration approximately 10 times the method-stated or laboratory-determined method detection limit
___	C.1(c)	Are at least 4 such aliquots prepared & analyzed according to the test method Note: These analyses may occur either concurrently or over a period of days
___	C.1(d)	Does the laboratory calculate the mean recovery in the appropriate reporting units & the standard deviation of the population sample (n-1) in the same reporting units for each parameter of interest using all of the analysis results obtained Note: When it is not possible to assess mean & standard deviation, such as for presence-absence & logarithmic values, the laboratory must assess performance against established & documented criteria
___	C.1(e)	Are the mean and standard deviation for each parameter compared to the corresponding acceptance criteria for precision & accuracy in the test method (if applicable) or in laboratory-generated acceptance criteria (if the method or analyte is non-standard)
___	C.1(e) D.1.3(a)	Does the laboratory not begin the analysis of actual samples until the acceptance criteria are met for all parameters of interest
___	C.1(f)	When one or more parameters fail at least one of the acceptance criteria, does the analyst: - Locate & correct the source of the problem, then repeat the test for all parameters of interest, OR - Repeat the test for all parameters that failed to meet criteria Note: Repeated failure from employing the second option above indicates a general problem with the entire measurement system, and the analyst must then perform the first option above
___	C.1	Does the laboratory document in its Quality Manual other adequate approaches to assessing Demonstration of Capability if the above procedure is not required by the mandated test method or regulation and if the laboratory elects not to perform the above procedure
___	C.2	Does the laboratory use a certification statement to document the completion of each initial Demonstration of Capability
___	C.2	Are copies of these certification statements retained in the personnel files of each affected employee

COMMENTS: If applicable, list all analytes for the above test method where the above Standards are not being met.

TEST METHOD ASSESSED _____

___	5.6.2(c)(4) 5.10.2.1(b)	Does the laboratory require continued demonstration of method performance (by each Analyst at least once per year)
___	5.10.2.1(c)	Does the laboratory complete, retain, & make available for review an appropriate certification statement documenting each Demonstration of Capability (initial or continuing)
___	5.10.2.1(c)	Does the laboratory retain all associated supporting data necessary to reproduce the analytical results summarized in the Demonstration of Capability checklists
___	5.10.2.1(d) D.1.3(a)	Does the laboratory complete a demonstration of capability each time there is a change in instrument type, personnel, or test method
___	5.10.2.1(e)	Does the laboratory fully document the achievement of demonstration of capability requirements for each specialized work cell **Note:** A work cell is defined as a group of analysts with specifically defined tasks that together perform the test method **Note:** Each Analyst must demonstrate capability for each analyte & test method (e.g. EPA 3510 & 3540, or EPA 8100 & 8270); however, under the "work cell" concept, demonstration of capability is not needed for each permutation or combination of preparation method & analysis method i.e. A Demonstration of Capability where a first Analyst extracts PAH's by EPA 3510, a second Analyst determines the PAH's by EPA 8270, and each PAH meets acceptance criteria for precision & accuracy validates the first Analyst for all PAH sample preparations by EPA 3510 (regardless of determinative method) and the second Analyst for PAH analyses by EPA 8270 (regardless of sample preparation method) (The first Analyst & the second Analyst constitute the "work cell" for this example)
___	5.10.2.1(f)	Does the laboratory demonstrate & document acceptable performance through acceptable continuing performance checks (e.g laboratory control samples) each time that membership in a work cell changes
___	5.10.2.1(f)	Do the new members of the work cell work with experienced analysts in the specialty area
___	5.10.2.1(f)	Does the laboratory repeat a Demonstration of Capability with the new work cell if the first 4 continuing performance checks following the change in personnel produce a failure in any sample batch acceptance criteria
___	5.10.2.1(f)	Does the laboratory repeat a Demonstration of Capability if the entire work cell is changed or replaced
___	5.10.2.1(g)	Is the performance of the work cell as a group linked to the training records of the individual members of the work cell
___	5.1(b)	Does the laboratory's acceptance criteria for precision & accuracy fulfill the requirements in the mandated test method **Note:** See page 45 for such Demonstration of Capability acceptance criteria

COMMENTS: If applicable, list all analytes for the above test method where the above Standards are not being met.

TEST METHOD ASSESSED: _____

___	D	Does the laboratory assess & evaluate all quality control measures on an on-going basis
___	D	Does the laboratory use quality control acceptance criteria to determine the validity of the data Note: Acceptance criteria from method blanks (D.1.1(a)) & laboratory control samples (D.1.1(b)(1)) are the minimum criteria to be used to determine batch acceptance
___	D.1.1(a)	Does the laboratory analyze method blanks at a frequency of at least one per sample batch per matrix type per sample extraction or preparation method
___	D.1.1(a)	Does the laboratory investigate the source of contamination & take measures to correct, minimize, or eliminate the problem: - If the method blank contamination exceeds a concentration greater than 1/10 of the measured concentration of any sample in the associated sample batch OR - If the method blank contamination exceeds the concentration present in the samples & is greater than 1/10 of the specified regulatory limit
___	D.1.1(a)	Are all samples associated with a contaminated blank reprocessed for analysis or reported with appropriate data qualifying codes
___	D.1.1(b)(1)	Does the laboratory analyze at least one laboratory control sample (QC Check Sample) per batch of 20 or less samples per matrix type per sample extraction or preparation method Note: This Standard does not apply to analytes for which spiking solutions are not available (e.g. Total Suspended Solids, Total Dissolved Solids, Total Volatile Solids, Total Solids, pH, Color, Odor, Temperature, Dissolved Oxygen, or Turbidity) Note: The matrix spike may be used in place of this control sample as long as the acceptance criteria are as stringent as for the laboratory control sample
___	D.1.1(b)(2)	Does the laboratory perform matrix spikes at a frequency of at least one in 20 samples per matrix type per sample extraction or preparation method Note: This Standard does not apply to analytes for which spiking solutions are not available (e.g. Total Suspended Solids, Total Dissolved Solids, Total Volatile Solids, Total Solids, pH, Color, Odor, Temperature, Dissolved Oxygen, or Turbidity)
___	D.1.1(b)(2)	Does the laboratory rotate the selected sample(s) for matrix spikes among client samples so that various matrix problems may be noted and/or assessed
___	D.1.1(b)(2)	Does the laboratory report poor performance in a matrix spike to the client whose sample was used for the spike
___	D.1.1(b)(3)	Does the laboratory add surrogate compounds to all samples, standards, & blanks for all organic chromatography methods Note: This Standard does not apply if the sample matrix precludes the use of surrogates or when a surrogate is not available
___	D.1.1(b)(3)	Does the laboratory report poor surrogate recovery to the client whose sample produced the poor recovery

TEST METHOD ASSESSED: _____

___	D.1.1(b)(4)	If the test method does not specify the spiking compounds, does the laboratory spike all reportable components in the laboratory control sample & the matrix spike Note: A representative number (10%) of the listed components may be used in the spike in cases: - Where components are incompatible. - Where the test method has an extremely long list of components. OR - Where the components interfere with accurate assessment (e.g. simultaneously spiking chlordane, toxaphene, & PCB's; simultaneously spiking primary amines & ketones)
___	D.1.1(b)(4)	Do the selected components of each spiking mix represent all chemistries, elution patterns, & masses
___	D.1.1(b)(4)	Do the selected components of each spiking mix include permit-specified analytes & other client requested components
___	D.1.1(b)(4)	Has the laboratory used all reported components in the spike mixture within a 2-year time period
___	D.1.1(b)(4)	Has the laboratory ensured that no one component or components dominate the spike mixture
___	D.1.2	Does the laboratory analyze matrix spike duplicates or laboratory duplicates at a minimum frequency of one in 20 samples per matrix type per sample extraction or preparation method
___	D.1.2	Does the laboratory document its procedure for selecting the use of the appropriate type of duplicate
___	D.1.2	Is the selected sample for the duplicate rotated among client samples so that various matrix problems may be noted and/or addressed
___	D.1.2	Does the laboratory report poor performance in a duplicate to the client whose sample was used for the duplicate
___	5.1(b)	Does the laboratory's acceptance criteria for blanks, laboratory control samples, duplicates, & matrix spikes fulfill the requirements in mandated test methods Note: See pages 46-48 for acceptance criteria
___	5.1(b)	Does the laboratory fulfill additional requirements specified in the mandated test method or regulation Note: See pages 50-52 for the additional requirements stated in test methods

COMMENTS: List analytes & NELAC Standards where the above requirements are not being fulfilled

CHEMISTRY TEST METHOD EVALUATED: _____

____ D.1.4 Has the laboratory determined the detection limit(s) by the protocol in the mandated test method or applicable regulation

Note: An example is the Method Detection Limit determination under SDWA & CWA

Note: If the protocol for determining detection limits is not specified, the laboratory must still determine the detection limit(s) but according to a procedure that reflects instrument limitations & intended application of the test method

Note: The detection limit study is not required for components for which spiking solutions or quality control samples are not available (e.g. odor & temperature)

____ D.1.4(b) Has the laboratory initially determined the detection limits for the compounds of interest in this test method in a matrix in which there are no target analytes or interferences at a concentration that would impact the results

Note: If this is not possible, the laboratory must determine these detection limits in the matrix of interest

____ D.1.4(c) Does the laboratory determine detection limits each time there is a significant change in the test method or instrument type

____ D.1.4(d) Does the laboratory include all sample processing steps in the analytical method in the determination of the detection limit

____ D.1.4(e) Has the laboratory documented all procedures used in the determination of the detection limit

Note: This documentation must include the matrix type

____ D.1.4(e) Has the laboratory retained all supporting data used in the determination of the detection limit

____ 5.1(b) Do the laboratory's detection limits fulfill the requirements of mandated test methods or regulations (i.e. reliably & consistently below any SDWA Maximum Contaminant Levels, RCRA Toxicity Characteristics, CWA effluent limitations, or other concentration levels upon which regulatory decisions are based)

Note: See page 49 for SDWA Maximum Contaminant Levels & RCRA Toxicity Characteristics

Note: Other regulations (including state regulations) & permits may contain additional requirements for Reporting Limits, Minimum Levels, Lower Limits of Detection, & other criteria

COMMENTS: List analytes for which the above Standards or requirements for measurement sensitivity have not been fulfilled

CHEMISTRY TEST METHOD EVALUATED: _____

Note: These Standards apply only to chromatography methods

___ D.1.7(a) Has the laboratory developed & documented acceptance criteria for retention time windows

___ D.1.7(b) Does the laboratory perform confirmations to verify compound identification when positive results are detected on a sample from a location that has not been previously tested by the laboratory

Note: These confirmations are performed on pesticides, herbicides, acid extractables, or other organic tests, or when recommended by the analytical test method

Note: Confirmation is not required when the analysis involves the use of a mass spectrometer

Note: Confirmation is required unless stipulated in writing by the client

___ D.1.7(b) Does the laboratory document all confirmations of compound identity

___ D.1.7(c) If a mass spectrometer is used, has the laboratory documented acceptance criteria for mass spectral tuning

COMMENTS:

PERFORMANCE-BASED MEASUREMENT SYSTEM TEST METHODS

Note: In addition to the Standards assessed for each mandated, approved, or generally-accepted test method, complete the following checklist for each **non-independently validated, non-approved, or laboratory-developed test method**

CHEMISTRY TEST METHOD EVALUATED (reference): _____

____ 5.10.2(a)(2) Is the laboratory's test method developed under the Performance-Based Measurement Systems (PBMS) approach **fully documented & validated**

____ 5.10.2(a)(2) Does the laboratory make its PBMS test method **available to the client & other recipients** of the relevant reports

COMMENTS: Use this space to records details of the laboratory-developed PBMS Test Method, including the following:

- target analytes, concentration ranges, applicable sample matrices & types
- analysis technique, plus any sample preparation procedures
- required sample collection, preservation, & handling requirements
- reagent purity, preparation, storage, & shelf-life requirements
- acceptance criteria for calibrations, verifications, accuracy, precision, sensitivity
- interferences affecting analyte selectivity & specificity, plus procedures to minimize them
- equipment & supplies required
- standards required (including traceability to national standards, preparation procedure & frequency)
- quality control procedures required, frequency, criteria for data acceptability
- any safety precautions required
- data calculations, reductions, or statistical evaluations needed
- method performance certifications for analysts, plus basis for such certification
- method validation procedure, references, & participants

Such information, including the method number reference, must be reported to the NELAC Board of Directors and may be critical for acceptance by a Secondary Accrediting Authority

ATTACHMENT 2
Internal Audit Report

SWLO/AATS
QA DEPT.
INTERNAL AUDIT REPORT
AUDIT DATE: 06/05/98
AUDITOR: [illegible]

DISCUSSION:

An internal audit was performed regarding our analysis of Ignitability. The Analytical procedure was compared to our Standard Operating Procedure (SWL-IN-703), SW-846 Method 1010. and ASTM procedure D93-97.

FINDINGS:

The analyst (Steve Southard) explained the process of pouring the sample into the test cup while still cold from storeage into the ignition cup. He was questioned about making remarks about samples in plastic containers or containers with leaks. etc.; he mentioned that he could write those items into the comments section of his log. The flash was checked at every two degrees F which meets & exceeds the SOP & Method. The p-Xylene is used as the calibration standard; however. sometimes the sample ignites below the analyst followed what was ing attachment shows a comparison between the procedure being performed / our Standard Operating Procedure / and the Method . Significant differences are listed.

NONCONFORMANCE REPORTS NEEDED: - Although it is not spelled out in the SOP. action should be taken if/when p-Xylene checks are outside of performance listed in table 1.

CORRECTIVE ACTIONS NEEDED: - SOP needs to be revised to bring in line with ASTM procedure D93.

ATTACHMENT 3
Technical Audit Schedules

Internal
Technical Audit Schedule

Month	Section(s)
January	Inorganics - Wet Chem
February	Extractions
March	GC/HPLC
April	SVOA/DIOXIN
May	VOA / AIR
June	RAD
July	Inorganics - Metals
August	Extractions
September	GC/HPLC
October	SVOA/Dioxin
November	VOA / AIR
December	RAD

ATTACHMENT 4
System Audit Schedules

Internal
Systems Audit Schedule

SOP	SYSTEM	JAN	FEB	MAR	APR	MAY	JUN	JUL	AUG	SEPT	OCT	NOV	DEC
GA101	**DOC CONTROL**										X		
GA132	DATA REPORTING (NON-EPA)										X		
GA106	ARCHIVAL										X		
GA131	**PROJECT MANAGEMENT**			X									
GA102	**INFO SYSTEMS**				X								
GA138	LIMS BACK-UP				X								
GA139	LIMS MAINTENANCE/MNGMNT				X								
GA104	**TRAINING**	X											
GA107	**PROCUREMENT**	X											
GA108	STD TRACEABILITY	X											
GA127	PREV. MAINTENANCE	X											
GA111	**LAB SAFETY PLAN**	X	X	X	X	X	X	X	X	X	X	X	X
GA123	CONTINGENCY PLAN							X					
GA130	RESP. PROTECTION PLAN	X	X	X	X	X	X	X	X	X	X	X	X
GA114	HAZ WASTE			X			X			X			X
GA112	**CONTROL CHARTS**					X							
GA105	CORRECTIVE ACTION					X							
GA113	DET. LIMITS					X							
GA115	**SAMPL CUSTODIAN**		X										
GA124	LIMS SMPL REC.		X										
GA135	SAMPL LOG-IN REVIEW		X										
GA110	SAMPLE CUSTODY		X										
GA116	BOTTLE PREP		X										
GA128	**ZONE**	X	X	X	X	X	X	X	X	X	X	X	X
GA125	LAB WATER SUPPLY	X											
GA120	BALANCE CHECKS	X	X			X	X		X	X		X	X
GA121	GLASSWARE PREP	X	X					X	X				

SOUTHWEST LABORATORY OF OKLAHOMA, INC. *and*
AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.

TM

Standard Operating Procedure

Laboratory Documentation of Sample Custody

Document No.: SWL-GA-110

Rev No. / Date: Rev. 3.3 — 09/08/00

OFFICIAL COPY RED STAMP INDICATES ORIGINAL

APPROVALS	DATES
Chuck Homer Procedure Prepared By	9/11/00 Date
Arthur L. Maulls Laboratory Manager or Director	09/11/00 Date
Bryan Tuck Laboratory Safety Officer	9/11/2000 Date
Chuck Homer Laboratory QA/QC Officer	9/11/00 Date

(Effective Date is 5 calendar days after the last signature above - QA/QC Officer)

Document Status

[ ] Controlled DC No. _____ Issued to: _____ Date: _____ QA Initials: _____

[✓] Official Copy OC No.: 2431 Issued to: Linda Karsonovich Date: 11/14/00 QA Initials: _____

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LABORATORY DOCUMENTATION OF SAMPLE CUSTODY

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1.0 PURPOSE AND APPLICATION

A critical aspect of sound sample collection and analysis protocols is the maintenance of strict COC procedures. COC procedures include inventorying and documentation during sample collection, shipment, and laboratory processing. A sample is considered to be in an individual's custody if the sample is: (1) in the physical possession or view of the responsible party; (2) secured to prevent tampering; or (3) placed in a restricted area by the responsible party. The laboratory is responsible for this documentation of sample custody throughout the handling, processing, and analysis of samples.

2.0 RESPONSIBILITIES

2.1 Sample Custodian

2.1.1 Document that shipments were secured with tamper evident custody seals.

2.1.2 Accept custody of samples and initiate the internal chain of custody documentation

2.1.3 Oversees the Internal Chain of Custody, making sure that all required information is accurately documented.

2.1.4 Notifies the Sample Receiving Supervisor and/or Quality Assurance Officer when forms are not being filled out correctly.

2.2 Sample Preparation Technicians/Analytical Chemists

Documents acceptance and relinquishing of samples prior to returning samples to storage.

2.3 Sample Receiving Supervisor/QA Officer

Follows-up with any corrective action necessary, upon notification of deficient internal chain of custody documentation.

3.0 EQUIPMENT

3.1 LIMS Monitor/Keyboard Access

3.2 Printer (HP Laserjet4 or equivalent)

4.0 PRECAUTIONS

Not applicable.

5.0 PROCEDURE

5.1 Sample Label

5.1.1 Description — Field Sample Label. A label is attached to all sample containers at the time of collection. The label is written in indelible ink and contains the following information:

5.1.1.1 Sample number/identification

5.1.1.2 Date and time collected

5.1.1.3 Purpose of the sample (analyte and sample group)

5.1.1.4 Source/location and location of the sample

5.1.1.5 Contract task number and title of project

5.1.1.6 Preservative used (if any)

5.1.1.7 Collector's name or initials

5.1.2 An example of a sample label is presented in Attachment 1.

5.2 Chain of Custody Record — External COC

5.2.1 Description

5.2.1.1 Sample custody is initiated with the detailed record keeping by the field sampling personnel. COC establishes the documentation and control necessary to identify and trace a sample from sample collection to final analysis. It includes field sample labeling to prevent mix-up, tamper evident custody seals to prevent sample tampering, secure custody, and provide the recorded support information for potential litigation.

5.2.1.2 COC forms are used to document the integrity of all samples. To maintain a record of sample collection, transfer between personnel, shipment, and receipt by the laboratory, a COC form will be filled out for each sample set at each sampling location. The COC form will contain the following information:

- Sample number (for each sample in shipment)
- Collection date (for each sample shipment)
- Time sample was obtained/or collected
- Number of containers of each sample
- Sample description (environmental matrix)
- Analyses required for each sample
- Shipment number
- Shipping address of the laboratory
- Date, time and method of shipment
- Spaces to be signed as custody is transferred.

5.2.1.3 The individual in charge of shipping samples to the laboratory is also responsible for completing the COC form. This individual will also inspect the form for completeness and accuracy. Any changes made to the COC form shall be initialed by the person making the change. An example of the COC form is presented in Attachment 2.

5.3 Transfer of Custody and Shipment-External

5.3.1 Samples are to be accompanied by an approved COC record. When the possession of samples are transferred, the individual relinquishing the samples signs and records the date and time on the COC document. The individual receiving the samples repeats the procedure. This record represents the official documentation for all transference of the sample custody until samples have arrived at the laboratory.

5.3.2 If samples are to be split with another laboratory facility or governmental agency, a separate COC record is prepared for those samples. This COC record indicates with whom the samples have been split and is appropriately signed and dated with the time of transfer of splits.

5.4 Laboratory Custody Procedures-Internal

5.4.1 The Sample Control program describes the laboratory custody procedures associated with sample receipt, storage, preparation, analysis and security. Sample control is maintained at SWLO through the use of several tracking systems designed to protect sample integrity. Tracking systems include the use of laboratory COC procedures and sample analysis requests (in the form of worksheets), Internal Chain of Custody forms, the Laboratory Information Management System (LIMS), Laboratory log Books (Extraction, Digestion and Analytical Run Logs).

5.4.2 Sample Tracking

5.4.2.1 An overview of the sample tracking and Internal Chain of Custody, (ICOC) procedure to be employed is presented in the Attachment 3 flow diagram. It includes the following components:

- Laboratory COC documentation is initiated by the SC when the sample is relinquished by the courier.
- After sample shipment arrival, the SC begins sample inspection and log-in. All samples are inspected: comparisons are made between the clients' paperwork and that paperwork supplied by the Project Officer. Anomalies are noted and resolved with the client.
- Suspected Radioactive samples are handled and "screened" by the Radiation Safety Officer or Designee as per SOP, SWL-RD139 in the Radiation Control Area.
- Each sample is assigned a unique SWLO laboratory identification number, which is cross-coded with the client's identification.

Sample identification information is entered into the computerized laboratory database, and the assigned number is used to track sample locations and status throughout the analytical process.

5.4.2.2 The following sample information is recorded into the computerized laboratory database system:

- Customer and project information
- Date of receipt
- Client identification
- Date sampled
- Matrix Type
- Number of containers
- Analytical requirements
- Other pertinent comments
- Radiation Screening results (RAD samples only)

5.4.2.3 The SC logs in samples with the tests and test code information supplied by the project officer.

5.4.2.4 The field COC document is completed and copies returned to the appropriate party(s).

5.4.2.5 After the sample is logged in, an Internal COC form (see Attachment 4) is generated. A facility transfer sheet (Attachment 5) is completed for those tests performed at one of our affiliated facilities. Analytical tests not performed by SWLO/AATS or affiliates (i.e. physical parameters such as Porosivity, Bulk Density, and Atterburg Limits), are transferred using an Outside Analytical Services request Form (Attachment 6).

5.4.2.6 This ICOC (Attachment 4) documents the movement of the sample within the laboratory from storage to sample preparation and back to sample storage.

5.4.2.7 When all analyses are complete, ICOC documents are filed with appropriate data in the report file.

5.4.2.8 Samples that are depleted with the analytical process are recorded on the depleted bottle log (Attachment 8). The remaining sample volumes are archived. A monthly archive list is generated from LIMS (Attachment 7) samples and extracts are moved into Archive following report generation and stored for approximately 90 days (1yr. for extracts). Following the archive period, samples/extracts are disposed of. Records of disposal are maintained on the Sample Disposal Forms (see SWL-GA-114 Standard Operating Procedure for Hazardous Waste Management).

Drum numbers listed on the disposal forms are recorded on the manifest upon transportation to the waste incinerating company.

5.4.2.9 In addition to the depleted bottle log and Sample Disposal Forms the SWLO Sample Disposition Program is used which details where and when samples are disposed. Attachment 9 shows the screens available for these records. The program is available on the Sample Receiving and Hazardous Waste Management Systems. The responsibility of maintaining these records also lay with the Hazardous Waste Manager and the Sample Receiving Personnel.

5.4.2.10 In addition to the internal and external COC documents, a computer-generated listing of the sample analysis parameters is used to control sample flow and facilitate tracking within the laboratory. Each laboratory unit is given the list of parameters and is responsible for maintaining sample integrity (holding time), fulfilling COC requirements, scheduling sample flow, and tracking sample status.

6.0 RECORDS

All initial paperwork (i.e., Chain of Custody, Sample Log-In Forms: OE-1, cooler Receipt Forms, etc.

6.1 Field Chain of Custody's are maintained (after sample log-in), in either the reporting department files, the organic data assembly files or the inorganic data assembly files until reporting. Originals or copies are sent back to the client with the report.

6.2 Internal Chain of Custody sheets, used by analysts to sign samples in or out, are located outside the walk-in cooler storage. Upon completion, the Internal COC is incorporated into the case file.

7.0 REFERENCES

Not applicable.

8.0 DEFINITIONS

Not applicable.

9.0 ATTACHMENTS

See the following 8 Attachments.

ATTACHMENT 1
Example Custody Seal for Sample Containers/Coolers

SOUTHWEST LABORATORY OF OKLAHOMA, INC.
(918)251-2858

SITE NAME	DATE
ANALYSIS	TIME
	PRESERVATIVE

SPECIALTY CLEANED CONTAINER

SWLO
(918) 251-2858

Designate comp / grab	Time	mo/day/year	Station No.	Project Code
Samplers		Preserved yes / no	Station Loc.	Remarks
				Tag No.

Analyses

FIG. 3.1-A
Example of Sample Bottle Label (top) & Sample Tag (right).

CUSTODY SEAL
Date _____
Signature _____
Southwest Laboratory of Oklahoma

CUSTODY SEAL
Date _____
Signature
Southwest Laboratory of Oklahoma

ATTACHMENT 2
Chain of Custody Record

CHAIN OF CUSTODY RECORD

COC RECORD (110-ATT2 WB1) SWL-GA-110 Rev. 3.0 8/22/97 BOOK # GA-110-COC

SOUTHWEST LABORATORY OF OKLAHOMA, INC.
AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.
1700 W. Albany • Broken Arrow, Oklahoma 74012
Office: 918-251-2858 • Fax 918-251-2999

SAMPLING FIRM | CLIENT CONTACT | PHONE NUMBER

PROJECT NUMBER | PROJECT NAME

SAMPLER'S: (Signature)

ANALYTICAL TESTS REQUESTED

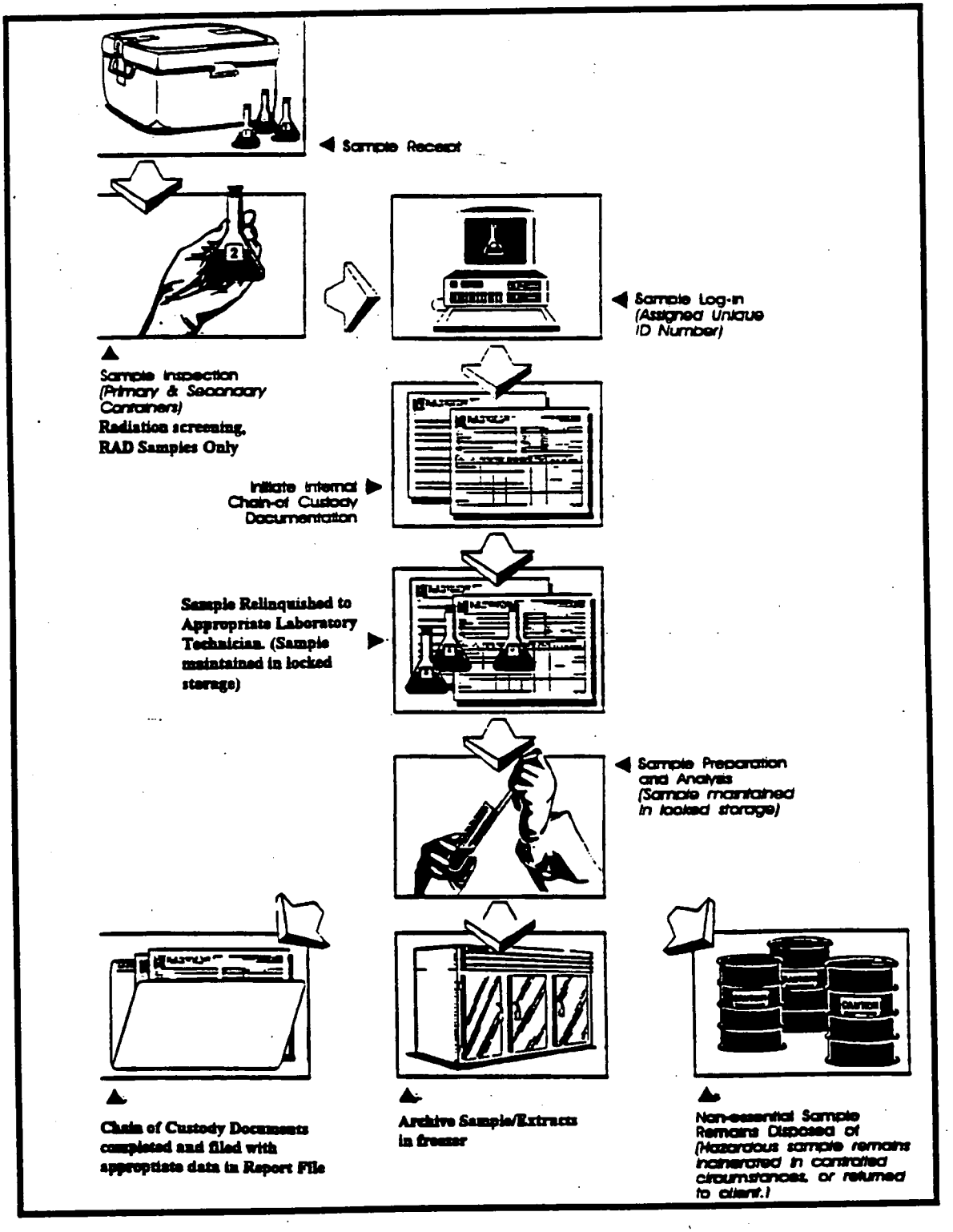
STA. NO	DATE	TIME	COMP.	GRAB	STATION LOCATION	MATRIX	NO OF CONTAINERS									REMARKS

RELINQUISHED BY: (Signature)	DATE	TIME	RECEIVED BY: (Signature)	RELINQUISHED BY: (Signature)	DATE	TIME	RECEIVED BY: (Signature)
RELINQUISHED BY: (Signature)	DATE	TIME	RECEIVED BY: (Signature)	RELINQUISHED BY: (Signature)	DATE	TIME	RECEIVED FOR LABORATORY BY: (Signature)
RELINQUISHED BY: (Signature)	DATE	TIME	RECEIVED BY: (Signature)	REMARKS:			

(SR012-0492-02)

Typist: LLR

ATTACHMENT 3
Chain of Custody Sample Tracking Flow Diagram



ATTACHMENT 4
Internal COC

Southwest Laboratory of Oklahoma
1700 W. Albany Suite C
Broken Arrow, OK 74012-1421

INTERNAL CHAIN OF CUSTODY

Date 08/25/2000
Episode 44091
Client
Project
Received 08/25/00

Test Code	Cont ID	#	#	#	#	#	#	#	Date/Time Out	Analyst	Date/Time In
		(Circle the Sample Numbers Checked out)									
GC340	C	1	2	3	4	6	7	8	8/29 8:00	GS	8/28 1:00
		9	10	11	12						
GC345	B	1	2	3	4	6	7	8			
		9	10	11	12						
IN660	A	1	2	3	4	5	6	7			
		8	9	10	11	12			8/26/00 0700	SR	8/26/00 1140
IN660	B	1	2	3	4	6	7	8			
		9	10	11	12						
IN660	C	1	2	3	4	6	7	8			
		9	10	11	12						
MS306	A	1	2	3	4	5	6	7			
		8	9	10	11	12	13				
MS306	B	13									
MS399	A	1	2	3	4	5	6	7			
		8	9	10	11	12	13		8/24/1400	SR	8/19 1450
MS399	B	13									
MS506	C	1	2	3	4	6	7	8			
		9	10	11	12						
MS599	C	1	2	3	4	6	7	8			
		9	10	11	12						
MT065	C	1	2	3	4	6	7	8			
		9	10	11	12						
MT185	C	1	2	3	4	6	7	8			
		9	10	11	12						
MT310	C	1	2	3	4	6	7	8			
		9	10	11	12						
MT600	C	1	2	3	4	6	7	8	8/28 945	AK	8/28 500
		9	10	11	12						
MT620	C	1	2	3	4	6	7	8	8/28 9:00	SR	8/28 500
		9	10	11	12						
MT800	C	1	2	3	4	6	7	8			
		9	10	11	12						
MT803	C	1	2	3	4	6	7	8			
		9	10	11	12						

Typist: LLR

ATTACHMENT 5
Facility Transfer Sheet

FACILITY TRANSFER SHEET

SAMPLE RECEIVING DEPARTMENT

ANALYTICAL SERVICES REQUEST

FACILITY TRANSFER SHEET (110-ATT5.WB1) SWL-GA-110 Rev. 3.0 8/22/97 BOOK # GA-110-FACLTRANS

Laboratory:	Date Results Required:
SWL Contact:	Comments:

SWL Sample #	Sample Matrix	No. of Containers	Testing Parameters							

Sample Matrix Key: *S = Soil* *SL = Sludge* *W = Water* *P = Petroleum* *O = Other*

CHAIN-OF-CUSTODY:

Relinquished by:	Date/Time	Received by:
Relinquished by:	Date/Time	Received for lab by:

SOUTHWEST LABORATORY OF OKLAHOMA, INC.
1700 W. ALBANY • BROKEN ARROW, OKLAHOMA 74012 • OFFICE (918) 251-2858 • FAX (918) 251-2599 [SR001-1093-02]

Typist: LLR

ATTACHMENT 6
Outside Analytical Services Request

OUTSIDE ANALYTICAL SERVICES REQUEST

SAMPLE RECEIVING DEPARTMENT

OUTSIDE ANALYTICAL SERVICES REQUEST (110-ATT6.WB1) SWL-GA-110 Rev 3.0 8/22/97 BOOK # GA-110-OUTANSRVREQ

TM

Laboratory:	Date Results Required:
SWL Contact:	Comments:

SWL Sample #	Sample Matrix	No. of Containers	Testing Parameters							

Sample Matrix Key: *S = Soil* *SL = Sludge* *W = Water* *P = Petroleum* *O = Other*

CHAIN-OF-CUSTODY:

Relinquished by:	Date/Time	Received by:
Relinquished by:	Date/Time	Received for lab by:

SOUTHWEST LABORATORY OF OKLAHOMA, INC.

1700 W. ALBANY • BROKEN ARROW, OKLAHOMA 74012 • OFFICE (918) 251-2858 • FAX (918) 251-2599 [SR018-1093-01]

ATTACHMENT 7
Archive Record Sheet

SOUTHWEST LABORATORY OF OKLAHOMA, INC.

ARCHIVE RECORD SHEET (110-ATT7.WB1) SWL-GA-110 Rev. 3.0 8/22/97 BOOK # GA-110-ARCHRECRD

Page 4

SAMPLES READY TO BE ARCHIVED

ARCHIVE DATE 08-29-97
DISPOSAL DATE 11-27-97

SAMPLE	CLIENT	DESCRIPTION	MATRIX	LOGIN
30295.01	AATS-E	CNK19 (CASE#25586)	W	07/25/97
30295.02	AATS-E	CNK20 (CASE#25586)	W	07/25/97
30295.03	AATS-E	CNK21 (CASE#25586)	W	07/25/97
30295.04	AATS-E	CNK23 (CASE#25586)	W	07/25/97
30295.05	AATS-E	CNK24 (CASE#25586)	S	07/25/97
30295.06	AATS-E	CNK25 (CASE#25586)	S	07/25/97
30295.07	AATS-E	CNK26 (CASE#25586)	S	07/25/97
30295.08	AATS-E	CNK28 (CASE#25586)	S	07/25/97
30295.09	AATS-E	CNK29 (CASE#25586)	W	07/25/97
30295.10	AATS-E	CNK30 (CASE#25586)	W	07/25/97
30295.11	AATS-E	CNK31 *QA/QC	W	07/25/97
30295.12	AATS-E	CNK32 (CASE#25586)	W	07/25/97
30295.13	AATS-E	CNK33 (CASE#25586)	W	07/25/97
30310.01	EPAMT137	MJP235 (CASE#25589)	S	07/25/97
30310.02	EPAMT137	MJP236 (CASE#25589)	S	07/25/97
30310.03	EPAMT137	MJP237 (CASE#25589)	S	07/25/97
30310.04	EPAMT137	MJP238 (CASE#25589)	S	07/25/97
30310.05	EPAMT137	MJP239 (CASE#25589)	S	07/25/97
30310.06	EPAMT137	MJP240 (CASE#25589)	S	07/25/97
30310.07	EPAMT137	MJP241 (CASE#25589)	S	07/25/97
30310.08	EPAMT137	MJP242 *QA/QC	S	07/25/97
30310.09	EPAMT137	MJP243 (CASE#25589)	S	07/25/97
30310.10	EPAMT137	MJP244 (CASE#25589)	W	07/25/97
30310.11	EPAMT137	MJP322 (CASE#25589)	S	07/25/97
30310.12	EPAMT137	MJP323 (CASE#25589)	S	07/25/97
30310.13	EPAMT137	MJP324 (CASE#25589)	S	07/25/97
30310.14	EPAMT137	MJP325 (CASE#25589)	S	07/25/97
30310.15	EPAMT137	MJP326 (CASE#25589)	S	07/25/97
30310.16	EPAMT137	MJP328 (CASE#25589)	S	07/25/97
30310.17	EPAMT137	MJP330 (CASE#25589)	S	07/25/97
30310.18	EPAMT137	MJP331 (CASE#25589)	S	07/25/97

ATTACHMENT 8
Depleted Bottle Log

Depleted Bottle Log

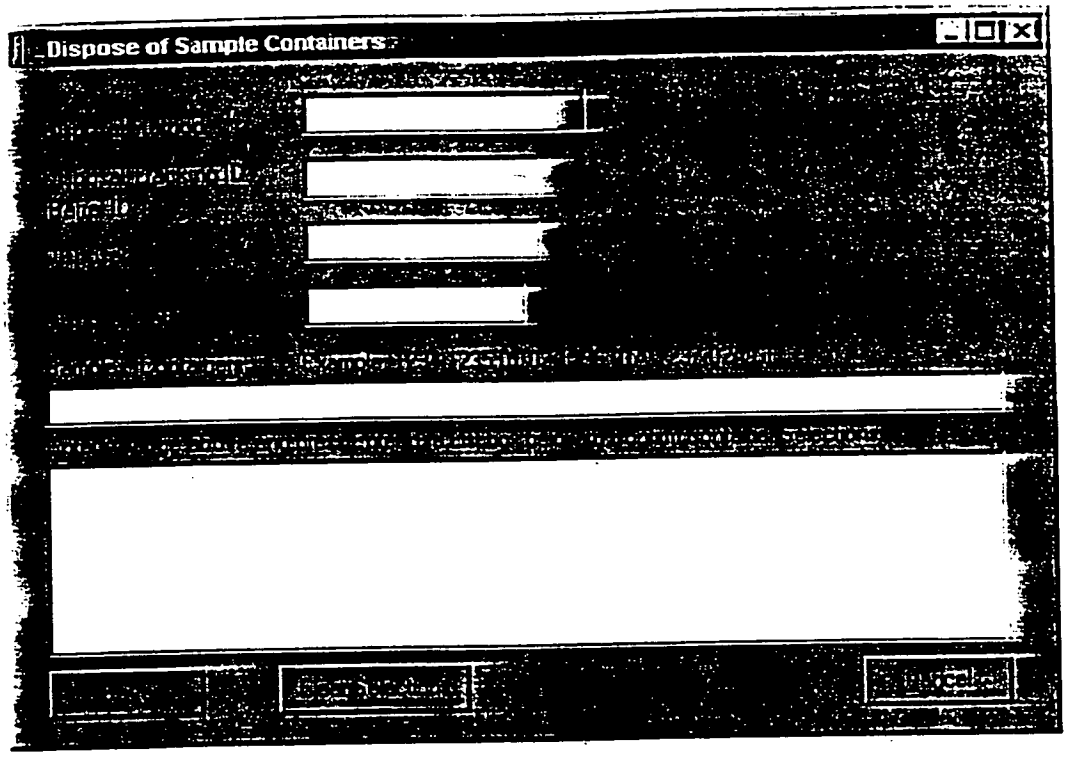
GENERAL ADMINISTRATIVE

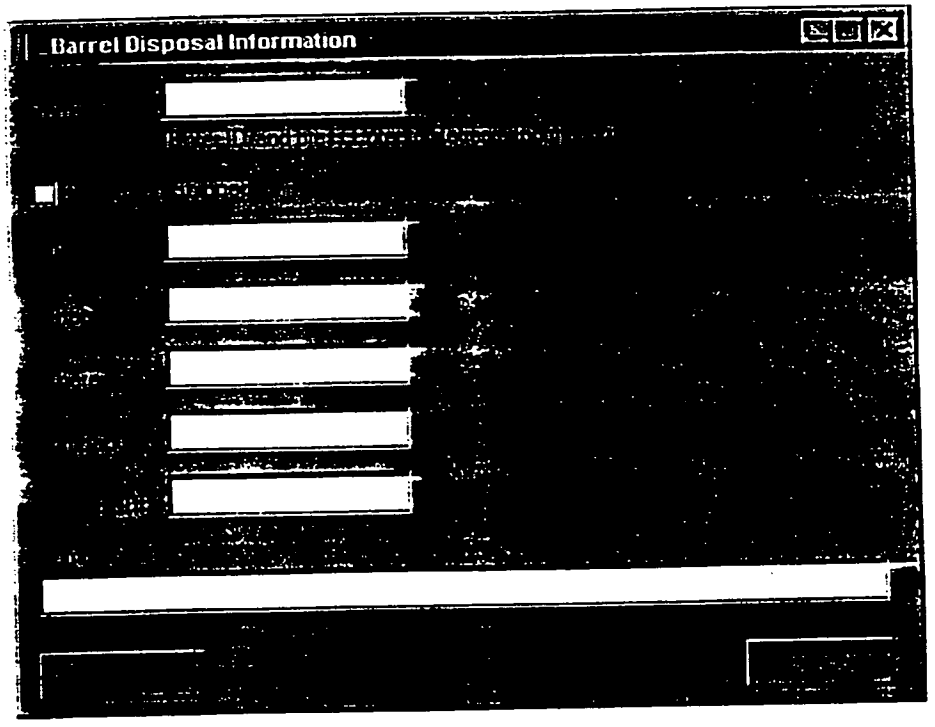
SOP SWL-GA-110, REV. # 3.2 FILE ID: GA110A8.doc Book # GA-110-PTCHBOTL

DATE	SAMPLE #	CLIENT	ANALYST

SOUTHWEST LABORATORY OF OKLAHOMA, INC.
1700 West Albany • Broken Arrow, OK 74012 • Office (918) 251-2858 • Fax (918) 251-2599

ATTACHMENT 9
Sample Disposition Program Window





SOUTHWEST LABORATORY OF OKLAHOMA, INC. *and*
AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.

TM

Standard Operating Procedure

Preventive Maintenance

Document No.: SWL-GA-127

Rev No. / Date: Rev. 4.0 — 7/31/00

OFFICIAL COPY RED STAMP INDICATES ORIGINAL

APPROVALS	DATES
Chuck Hoover Procedure Prepared By	7/31/00 Date
[illegible signature] Laboratory Manager or Director	08/02/00 Date
Bryan [illegible] Laboratory Safety Officer	8/2/2000 Date
Chuck Hoover Laboratory QA/QC Officer	7/31/00 Date

(Effective Date for is 5 calendar days after the last signature above - QA/QC Officer)

Document Status

[ ] Controlled DC No. _____ Issued to: _____ Date: _____ Initials: ____

[✓] Official Copy OC No.: 2431 Issued to: Linda Karsonovich Date: 11/14/00 Initials: [illegible]

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PREVENTIVE MAINTENANCE

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3.0 EQUIPMENT 1
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1.0 **PURPOSE AND APPLICATION.** Preventive maintenance is defined as an orderly program of positive actions for preventing failure of equipment and ensuring that the equipment is operating with the reliability required for quality results. The actions include specification checks, calibrating, cleaning, lubricating, reconditioning, adjusting and checking.

2.0 **RESPONSIBILITIES**

2.1 It is the responsibility of the analyst to ensure that the scheduled preventive maintenance is conducted.

2.2 It is the responsibility of the analytical supervisors/group-leaders/program managers to see that the analyst is adequately trained in the procedures of preventive maintenance and to aid the analyst when troubleshooting non-routine problems.

3.0 **EQUIPMENT.** All analytical instrumentation in the laboratory used in the generation of analytical results. (See section 5.2 of the *Laboratory Quality Assurance Plan*, SWL-GA-100).

4.0 **PRECAUTIONS**

4.1 See precautions in the owner's manuals for each instrument.

4.2 See the *Laboratory Safety Plan*, SWL-GA-111.

5.0 **PROCEDURE**

5.1 The laboratory has established the following preventive maintenance program:

5.1.1 Each type of equipment/instrument has a written Standard Operating Procedure (SOP), which describes the methods for routine inspection, cleaning, maintenance, testing, calibration, and/or standardization of the equipment. Instrument operating manuals are kept near the instrument or where analysts have easy access.

5.1.2 Analysts using the instruments are properly trained and develop trouble-shooting skills in equipment failure to reduce dependence upon outside servicing agencies. In complicated cases, the servicing agency or supplier is called to solve the problem. Instruments which are down, are tagged "out-of-service" (see Attachment 1), until they are brought back to analytical control. When the return to analytical control is complete, it is documented in the instrument's Maintenance Log.

5.1.3 Written equipment records are kept to document all inspection, maintenance, trouble-shooting, calibration, or modifications. Whenever maintenance is performed on an instrument, it is properly documented in a preventive maintenance logbook, which is kept near the equipment to monitor the adequacy of maintenance schedules. The records contain the date (month, day, year), description of the maintenance done, the actual findings, the name of the person doing the maintenance and a statement of whether the maintenance operations were routine, and if those operations followed the written SOP.

Typist: LLR

5.1.4 Performance criteria is established for judging when data from instrument performance checks indicate the need to make adjustments in the instrument operating conditions.

5.2 Chromatographic Instruments

5.2.1 Preventive maintenance is done through a daily performance check and calibration of standards. Parameters such as retention time and response factors are observed and back-checked with prior operational performance.

5.2.2 In addition, the following are done:

5.2.2.1 GC detectors are cleaned whenever performance degradation (i.e., calibration criteria are not met, retention time shifts, noisy baseline, etc.) is observed by the analyst.

5.2.2.2 Septa are replaced as needed.

5.2.2.3 Incoming gas drying cartridges are changed whenever the color of the adsorbent is noticed.

5.2.2.4 Effluent adsorbent traps are changed as needed.

5.2.2.5 Columns (GC and HPLC) are checked by performance and operating conditions when in use or prior to use.

5.2.2.6 Oven performance checked daily prior to use.

5.2.2.7 Details regarding maintenance of the GC's and HPLC's can be found in SWL-OP-201, SWL-OL-103, SWL-OG-114 and SWL-OG-118.

5.3 Gas Chromatography/Mass Spectroscopy (GC/MS)

5.3.1 The preventive maintenance includes:

Maintenance Requirements	Frequency
Clean filters on cooling fans	Monthly
Check cooling fan for proper operation	Monthly
Check cool-flow level	Monthly
Change mechanical pump oil	Every 6 months
Clean source and rods	As needed
Check power supplies in QEM Box	As needed
Clean printer inside and outside	As needed
General cleaning of instrument	As needed
All items from monthly maintenance schedule	As needed
Sensitivity analysis through BFB and DFTPP tune criteria	Every 12-hour clock

5.3.2 Additional details regarding preventive maintenance can be found in SWL-OV-105, SWL-OD-102, and SWL-OA-103.

5.4 Atomic Absorption Spectrophotometers/ICP

5.4.1 Preventive maintenance is done for atomic absorption through the following checks:

5.4.1.1 Minimum warm-up period of 30 minutes

5.4.1.2 Alignment of hollow cathode tube to produce the maximum emitted light to the detector

5.4.1.3 For flameless AA, the inert gas flow inside the furnace is optimized to ensure maximum sensitivity.

5.4.1.4 Digital readout values obtained for the standard curve of each element are checked to ensure linearity.

5.4.1.5 If readings are low, the operator checks the gas flows, burner or cell alignment, wavelength, slit width, photomultiplier voltage and lamp intensity prior to analysis

5.4.2 Details on the preventive maintenance for ICP and AA's can be found in SWL-IN-103.

5.5 General Laboratory Equipment

5.5.1 Analytical balances of various capacities and operational modes are calibrated semi-annually by a licensed specialist and officially recorded as verification of performance. Balances are checked with standard Class 1 calibration weights before use.

5.5.2 The pH/specific-ion meters are calibrated before use with a minimum of two standard solutions. All combination pH electrodes will be stored in pH 4 buffer solutions.

5.5.3 TCLP Extraction Equipment shall be checked as follows:

- Rotary Extractor-RPM checked monthly (28-32 rpm)
- ZHE's-Viton O-rings checked prior to use; replaced if worn and or ZHE has pressure problems.
- 2L Extraction vessels-check lip for cracks (upon use).
- Teflon Liner on lids-check for (cracks/tears) upon use.

5.5.4 Other inorganic preventive maintenance procedures can be found in SWL-IN-103.

6.0 RECORDS

6.1 Maintenance documentation is recorded in the Instrument Maintenance Log.

6.2 Details of these logs can be found in the following standard operating procedures:

SWL-IN-103	Inorganic Preventive Maintenance Schedule
SWL-OA-104	Air Logbook Maintenance
SWL-OD-102	Dioxin Logbook Maintenance
SWL-OG-114	GC Volatiles Logbook Maintenance
SWL-OL-103	Liquid Chromatography Instrument Maintenance Log
SWL-OP-201	Preventive Maintenance of Gas Chromatographs
SWL-OV-102	Volatile Logbook Maintenance
SWL-OV-105	Purge and Trap Maintenance

7.0 REFERENCES. N/A

8.0 ATTACHMENTS.

8.1 Equipment Out-of-Order

9.0 DEFINITIONS

9.1 GC..... Gas Chromatograph

9.2 HPLC ..... High Performance Liquid Chromatograph

9.3 ICP ..... Inductively Coupled Argon Plasma

9.4 AA..... Atomic Absorption

9.5 TCLP..... Toxicity Characteristic Leaching Procedure

9.6 ZHE..... Zero Headspace Extractor

ATTACHMENT 1

EQUIPMENT OUT-OF-ORDER SIGN

SOUTHWEST LABORATORY OF OKLAHOMA, INC.
AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.

TM

Equipment Out-of Order

Instrument ID: HP 5890 Series II GC

Serial No. 2950A27577

QCFORMS on 'SWLO1'/ORGANICS/out-of-Order.doc

SOUTHWEST LABORATORY OF OKLAHOMA, INC. *and*
AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.

TM

Standard Operating Procedure

Maintenance Of "Zone" Log Books

Document No.: SWL-GA-128

Rev No. / Date: Rev. 3.2 — 02/22/00

OFFICIAL COPY RED STAMP INDICATES ORIGINAL

APPROVALS	DATES
Chuck Hoover Procedure Prepared By	4/29/00 Date
[signature] Laboratory Manager or Director	05/01/00 Date
Bryan Hicks Laboratory Safety Officer	5/01/2000 Date
Chuck Hoover Laboratory QA/QC Officer	4/29/00 Date

(Effective Date is 5 calendar days after the last signature above - QA/QC Officer)

Document Status

[ ] Controlled DC No. _____ Issued to: _____ Date: _____ QA Initials: _____

[✓] Official Copy OC No.: 2431 Issued to: Linda Kassenovich Date: 11/14/00 QA Initials: CH

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MAINTENANCE OF "ZONE" LOG BOOKS

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6.3 Form GA128F3 "Oven Temperature Record" 2
6.4 Form GA128F4 "D.I. Water Conductivity Check" 2
6.5 Form GA128F5 "Daily Balance Checks" 2
6.6 Form GA128F6 "Fume Hood Daily Flow Check" 2
6.7 Form GA128F7 "Equalization Tank pH Check" 2
6.8 Form GA128F8 "Water Bath Monitoring Record" 2
6.9 Form GA128F9 "Sonicator Record" 2
6.10 Form GA128F10 "Sample Receipt Thermometer Calibration Check 2
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Attachment 7 Equalization Tank Weekly pH Check 10

Attachment 8 Water Bath Monitoring Record 11
Attachment 9 Sonicator Record 12
Attachment 10 Sample Receipt Thermometer Calibration Check 13
Attachment 11 pH Meter Calibration Log 14
Attachment 12 Hot Plate Monitoring Log 15

1.0 PURPOSE AND APPLICATION — The monitoring of temperatures (refrigerator/ovens) balances and fume hoods will be conducted daily, equalization tanks will be conducted after each use. Records will be maintained in a "Zone" logbook.

2.0 RESPONSIBILITIES — The following personnel are responsible in seeing that the Zone Logbooks are being maintained:

- SVOA – GC/MS (OS) — Harry Borg/Ann Wade
- GC Pesticides (OP) — Drew Cowan/Jeff Cummins
- HPLC (OL) — Jon Barrows
- RMCA (RD) — Kert Surface/David Hayes
- Volatile GC/MS (OV) — Gina Jackson/Janet Williamson
- Organic Extractions (OE) — Desmond Foster/Otto Simpson
- Sample Receiving (SR) —Jason Spriggs/Don Willis
- Inorganics/Metals (IN) — Deborah Inman/Susan Turner
- Air/Dioxin (OA/OD) — Jayant Shringarpure/Jim Whitaker
- GC-VOA (OG) — Matt Persons/Bryon Landtroop

The logs will be maintained by designated employees and reviewed by the above supervisors. An audit check will be done on all new zone log books prior to their release.

3.0 EQUIPMENT — Each zone will have its own logbook which will contain the forms for monitoring oven/refrigerator/freezer temperatures; balance check; fume hood air flow and D.I. water checks, and waste equalization tanks. Equalization tanks will be performed only in the radiation containment and metals areas.

4.0 PRECAUTIONS — All monitoring personnel will follow safety precautions as stated in SWL-GA-111, "*Laboratory Safety Plan*".

5.0 PROCEDURE

5.1 The laboratory is divided into the following zones:

- OS — SVOA-GC/MS
- OP — GC/Pesticides
- RD — Radioactive Materials Control Area (RMCA)
- OV — Voa-GC/MS
- OE — Organic Extractions
- SR — Sample Receiving
- IN — Wet Chemistry/ Metals
- OA — Air
- OD — Dioxin
- OG — GC Voa
- OL — HPLC

5.2 Details of how these logs are to be filled out are maintained in the following SOPs:

5.2.1 SWL-GA-128 — "*Maintenance of "ZONE" Log Books*"

5.2.2 SWL-GA-120 — "*Calibration of Electronic Balances*"

5.2.3 SWL-GA-111 — "*Laboratory Safety Plan*"

5.2.4 SWL-GA-114 — "*Hazardous Waste Management Plan*"

6.0 RECORDS

6.1 Form GA128F1 "Refrigerator Temperature Record" shall be maintained in a bound zone logbook in each laboratory area, which requires refrigerator temperature monitoring.

6.2 Form GA128F2 "Freezer Temperature Record" shall be maintained in a bound zone logbook in each laboratory area, which requires freezer temperature monitoring.

6.3 Form GA128F3 "Oven Temperature Record" shall be maintained in a bound zone logbook in each laboratory area, which requires oven temperature monitoring.

6.4 Form GA128F4 "D.I. Water Conductivity Check" shall be maintained in a bound zone logbook in each laboratory area, which requires the monitoring of D.I. water conductivity.

6.5 Form GA128F5 "Daily Balance Checks" shall be maintained in a bound zone logbook in each laboratory area, which requires the calibration of balances.

6.6 Form GA128F6 "Fume Hood Daily Flow Check" shall be maintained in a bound zone logbook in each laboratory area which requires the monitoring of fume hood velocities.

6.7 Form GA128F7 "Equalization Tank Weekly pH Check" shall be maintained in a bound zone logbook in the metals and radiation materials control area.

6.8 Form GA128F8 "Water Bath Monitoring Record" shall be maintained in a bound zone logbook in each laboratory area, which requires the monitoring of water bath temperatures.

6.9 Form GA128F9 "Sonicator Record" shall be maintained in a bound Zone Log Book in each laboratory area which requires calibration checks for sonicators.

6.10 Form GA128F10 "Sample Receipt Thermometer Calibration Check" shall be maintained in a bound Zone Log Book for the Sample Receiving Area.

6.11 Form GA128F11 "pH Meter Calibration Log" shall be maintained in a bound Zone Log Book for Organics Extractions.

6.12 Form GA128F12 "Hotplate Monitoring Log"

7.0 REFERENCES — N/A

8.0 ATTACHMENTS

8.1 Attachment 1 — Refrigerator Temperature Record, GA128F1.

8.2 Attachment 2 — Freezer Temperature Record, GA128F2.

8.3 Attachment 3 — Oven Temperature Record, GA128F3.

8.4 Attachment 4 — D.I. Water Conductivity Check, GA128F4.

8.5 Attachment 5 — Daily Balance Checks, GA128F5.

8.6 Attachment 6 — Fume Hood Daily Flow Check, GA128F6.

8.7 Attachment 7 — Equalization Tank Weekly pH Check, GA128F7.

8.8 Attachment 8 — Water Bath Monitoring Record, GA128F8.

8.9 Attachment 9 — Sonicator, GA128F9.

8.10 Attachment 10 — Sample Receipt Thermometer Calibration Check, GA128F10.

8.11 Attachment 11 — pH Meter Calibration Log

8.12 Attachment 12 – Hot Plate Monitoring Log

9.0 DEFINITION

- Zone = A defined area of the laboratory.

ATTACHMENT 1
Refrigerator Temperature Record

Refrigerator Temperature Record, SWL-GA-128

GENERAL ADMINISTRATIVE

SOP SWL-GA-128, REV. # 3.0 Book ID: GA-128-ZoneRef-___ FILE ID: GA128F1.doc

REFRIG: _____
MONTH: _____
THERM. ID: _____

CONTROL LIMITS: 2 °C TO 6.0 °C

DATE	TIME	ANALYST	TEMP °C / °F	WITHIN LIMITS ?	CORRECTIVE ACTION * (IF NEEDED)
1				YES NO	
2				YES NO	
3				YES NO	
4				YES NO	
5				YES NO	
6				YES NO	
7				YES NO	
8				YES NO	
9				YES NO	
10				YES NO	
11				YES NO	
12				YES NO	
13				YES NO	
14				YES NO	
15				YES NO	
16				YES NO	
17				YES NO	
18				YES NO	
19				YES NO	
20				YES NO	
21				YES NO	
22				YES NO	
23				YES NO	
24				YES NO	
25				YES NO	
26				YES NO	
27				YES NO	
28				YES NO	
29				YES NO	
30				YES NO	
31				YES NO	

REPAIR SERVICE: Tobey's Heating & A/C • Ken Toby • 664-1166
OK Refrigeration CO • McIntosh Services • 836-6401

ATTACHMENT 2
Freezer Temperature Record

Freezer Temperature Record, SWL-GA-128

GENERAL ADMINISTRATIVE

SOP SWL-GA-128, REV. # 3.0 Book ID: GA-128-ZoneFreez-___ FILE ID: GA128F2.doc

FREEZ: _____

MONTH: _____

THERM. ID: _____

CONTROL LIMITS: -10 °C TO -20 °C

DATE	TIME	ANALYST	TEMP °C / °F	WITHIN LIMITS ?	CORRECTIVE ACTION * (IF NEEDED)
1				YES NO	
2				YES NO	
3				YES NO	
4				YES NO	
5				YES NO	
6				YES NO	
7				YES NO	
8				YES NO	
9				YES NO	
10				YES NO	
11				YES NO	
12				YES NO	
13				YES NO	
14				YES NO	
15				YES NO	
16				YES NO	
17				YES NO	
18				YES NO	
19				YES NO	
20				YES NO	
21				YES NO	
22				YES NO	
23				YES NO	
24				YES NO	
25				YES NO	
26				YES NO	
27				YES NO	
28				YES NO	
29				YES NO	
30				YES NO	
31				YES NO	

REPAIR SERVICE: Tobey's Heating & A/C • Ken Toby • 664-1166
OK Refrigeration CO • McIntosh Services • 836-6401

ATTACHMENT 3
Oven Temperature Record

Oven Temperature Record, SWL-GA-128

GENERAL ADMINISTRATIVE

SOP SWL-GA-128, REV. # 3.0 Book ID: GA-128-ZoneOven-___ FILE ID: GA128F3.doc

OVEN: _____

MONTH: _____

THERM. ID: _____

CONTROL LIMITS: _____°C TO _____°C

DATE	TIME	ANALYST	TEMP. °C	WITHIN LIMITS?	CORRECTIVE ACTION * (IF NEEDED)
1				YES NO	
2				YES NO	
3				YES NO	
4				YES NO	
5				YES NO	
6				YES NO	
7				YES NO	
8				YES NO	
9				YES NO	
10				YES NO	
11				YES NO	
12				YES NO	
13				YES NO	
14				YES NO	
15				YES NO	
16				YES NO	
17				YES NO	
18				YES NO	
19				YES NO	
20				YES NO	
21				YES NO	
22				YES NO	
23				YES NO	
24				YES NO	
25				YES NO	
26				YES NO	
27				YES NO	
28				YES NO	
29				YES NO	
30				YES NO	
31				YES NO	

REPAIR SERVICE: Tobey's Heating & A/C • Ken Toby • 664-1166
OK Refrigeration CO • McIntosh Services • 836-6401

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1700 West Albany • Broken Arrow, OK 74012 • Office (918) 251-2858 • Fax (918) 251-2599

ATTACHMENT 4
D. I. Water Conductivity Check

D.I. Water Conductivity Check, SWL-GA-128

GENERAL ADMINISTRATIVE

SOP SWL-GA-128 Rev.3.0 Book ID: GA-128-ZoneDI-___ File ID: GA128F4.doc

MONTH: ____

ACCEPTANCE: <1.0 UMHOS/CM

Date	Lab Area	Conductivity umhos/cm	Indicator Light?	Analyst	Corrective Action (If Needed)
1			ON / OFF		
2			ON / OFF		
3			ON / OFF		
4			ON / OFF		
5			ON / OFF		
6			ON / OFF		
7			ON / OFF		
8			ON / OFF		
9			ON / OFF		
10			ON / OFF		
11			ON / OFF		
12			ON / OFF		
3			ON / OFF		
14			ON / OFF		
15			ON / OFF		
16			ON / OFF		
17			ON / OFF		
18			ON / OFF		
19			ON / OFF		
20			ON / OFF		
21			ON / OFF		
22			ON / OFF		
23			ON / OFF		
24			ON / OFF		
25			ON / OFF		
26			ON / OFF		
27			ON / OFF		
28			ON / OFF		
29			ON / OFF		
30			ON / OFF		
31			ON / OFF		

TYPIST: LLR

ATTACHMENT 5
Daily Balance Checks

Daily Balance Ch[illegible]s, SWL-GA-128

MONTH ____

WT. SET USEL ____

SOP SWL-GA-128 Rev.3.0 Book ID: GA-128-ZoneBaln- _____ File ID: GA128F5

(2 Place) * ACCEPTANCE: <100g ± 0.01g, 100 – 200g - ± 0.02g, >200g - ± 0.03g

(4 Place) ^ ACCEPTANCE: 1.0g - ± 0.0001g, 10.0g - ± 0.0002g, 20.0g - ± 0.0002g, >50.0g - ± 0.0003g

DATE	LAB AREA	BALANCE ID	ANALYST	WGHT	OBSERVED READING	WGHT	OBSERVED READING	WGHT	OBSERVED READING	WGHT	OBSERVED READING
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											

TYPIST: LLR

* = Balances that read to 2 decimal points ^ = Balances that read to 4 decimal points

ATTACHMENT 6
Fume Hood Daily Flow Check

Fume Hood Daily Flow Check, SWL-GA-128

GENERAL ADMINISTRATIVE

SOP SWL-GA-128, Rev. 3.0 Book ID: GA-128-ZoneFume-3 File ID: GA128F6

MONTH: _____

ACCEPTANCE: 75–200 FPM

DATE	LAB	FUME HOOD NUMBER	FLOW CHECK (FULL FACE)	FLOW CHECK (6in. sash ht.)	ANALYST
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					

REPAIR SERVICE: Tobey's Heating & A/C • Ken Toby • 664-1166

TYPIST: LLR

SOUTHWEST LABORATORY OF OKLAHOMA, INC.
1700 West Albany • Broken Arrow, OK 74012 • Office (918) 251-2858 • Fax (918) 251-2599

ATTACHMENT 7
Equalization Tank Weekly pH Check

Equalization Tank Weekly pH Check, SWL-GA-128

GENERAL ADMINISTRATIVE

SOP SWL-GA-128, Rev. 3.0 Book ID: GA-128-ZoneTank-3 File ID: GA128F7

MONTH: _____

ACCEPTANCE: 3–1

DATE	LAB AREA	pH READING	ANALYST
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			
15			
16			
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28			
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30			
31			

TYPIST: LLR

ATTACHMENT 8
Water Bath Monitoring Record

Water Bath Monitoring Record, SWL-GA-128

GENERAL ADMINISTRATIVE

SOP SWL-GA-128, REV. # 3.0 Book ID: GA-128-ZoneBath-___ FILE ID: GA128F8.doc

MONTH: _____

THERM. ID: _____

CONTROL LIMITS: ____°C TO ____°C

DATE	TIME	ANALYST	BEG. TEMP °C/°F	END TEMP °C / °F	WITHIN LIMITS ?	CORRECTIVE ACTION * (IF NEEDED)
1					YES NO	
2					YES NO	
3					YES NO	
4					YES NO	
5					YES NO	
6					YES NO	
7					YES NO	
8					YES NO	
9					YES NO	
10					YES NO	
11					YES NO	
12					YES NO	
13					YES NO	
14					YES NO	
15					YES NO	
16					YES NO	
17					YES NO	
18					YES NO	
19					YES NO	
20					YES NO	
21					YES NO	
22					YES NO	
23					YES NO	
24					YES NO	
25					YES NO	
26					YES NO	
27					YES NO	
28					YES NO	
29					YES NO	
30					YES NO	
31					YES NO	

REPAIR SERVICE: Tobey's Heating & A/C • Ken Toby • 664-1166
OK Refrigeration CO • McIntosh Services • 836-6401

ATTACHMENT 9
Sonicator Record

SONICATOR RECORD, SWL-GA-128

GENERAL ADMINISTRATIVE

SOP SWL-GA-128, REV. # 3.0 Book ID: GA-128-ZoneSoni-___ FILE ID: GA128F9.doc

MONTH: _____

SONICATOR: A, B, C, E

CONTROL LIMITS: 17.5 (±3) DIV

DATE	TIME	ANALYST	WITHIN LIMITS ?	CORRECTIVE ACTION * (IF NEEDED)
1			YES NO	
2			YES NO	
3			YES NO	
4			YES NO	
5			YES NO	
6			YES NO	
7			YES NO	
8			YES NO	
9			YES NO	
10			YES NO	
11			YES NO	
12			YES NO	
13			YES NO	
14			YES NO	
15			YES NO	
16			YES NO	
17			YES NO	
18			YES NO	
19			YES NO	
20			YES NO	
21			YES NO	
22			YES NO	
23			YES NO	
24			YES NO	
25			YES NO	
26			YES NO	
27			YES NO	
28			YES NO	
29			YES NO	
30			YES NO	
31			YES NO	

ATTACHMENT 10
Sample Receipt Thermometer Calibration Check

Sample Receipt Thermometer Calibration Check, SWL-GA-128

GENERAL ADMINISTRATIVE

SOP SWL-GA-128, REV. # 3.0 Book ID: GA-128-ZoneTherm-___ FILE ID: GA128F10.doc

MONTH: _____

CONTROL LIMITS: ± 2 Degrees C.

DATE	S. R. Therm. I. D.	S. R. Therm. Reading	NIST Therm. I. D.	NIST Therm. Reading	S. R. Therm. Bias	S. R. Therm. Analyst	Corrective Action* (If Needed)
1							
2							
3							
4							
5							
6							
7							
8							
9							
10							
11							
12							
13							
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29							
30							
31							

ATTACHMENT 11
pH Meter Calibration Log

pH Meter Calibration Log, SWL-GA-128

SOP SWL-GA-128, REV. # 3.1 Book ID: GA-128-pHMeter-___ FILE ID: GA128F11.doc

MONTH: _____

CONTROL LIMITS: Slope ≥ 89.5

Date	pH buffer 4 Reading	pH buffer 7 Reading	pH buffer 10 Reading	Slope	Analyst	Comment
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
16						
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21						
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25						
26						
27						
28						
29						
30						
31						

ATTACHMENT 12
Hot Plate Monitoring Log

HOT PLATE MONITORING LOG

SOP SWL-GA-128, REV. 3.2 BOOK I.D.: GA-128-ZONEHP FILE I.D.: GA128F12

FOR THE MONTH OF: _____

1	2	3	4
5	6	7	8
9	10	11	12

	TEMP (degrees C) HOT PLATE #1	TEMP (degrees C) HOT PLATE #2	TEMP (degrees C) HOT PLATE #3	TEMP (degrees C) HOT PLATE #4	ANALYST	DATE
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						

Southwest Laboratory of Oklahoma, Inc. / American Technical & Analytical Services, Inc.
1700 W. Albany * Broken Arrow, Oklahoma 74011 * (918) 251-2858

SOUTHWEST LABORATORY OF OKLAHOMA, INC. *and*

AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.

TM

Standard Operating Procedure

Inorganic Data Package Assembly

Document No.: SWL-IN-102

Rev No. / Date: Rev. 3.2 — 04/07/00

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APPROVALS	DATES
Procedure Prepared By	4-7-00 Date
Program Manager or Section Supervisor	4-7-00 Date
Chuck Jones for Bryan Hicks Laboratory Safety Officer	4/7/00 Date
Chuck Jones Laboratory QA/QC Officer	4/7/00 Date

(Effective Date is 5 calendar days after the last signature above - QA/QC Officer)

Document Status

[ ] Controlled DC No. _____ Issued to: _____ Date: _____ QA: _____

[✓] Official Copy OC No.: 2382 Issued to: Tom Johnston Date: 05/05/00 QA: _____

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INORGANIC DATA PACKAGE ASSEMBLY

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1.0 PURPOSE AND APPLICATION.

1.1 The purpose of this Standard Operating Procedure is to describe the necessary forms and data required for an EPA CLP Package and a CLP-Like Package for non-EPA clients. The SOP will describe how the information on the forms is to be filled out and the proper order assemblage of the data package.

1.2 This procedure is applicable to all EPA CLP packages and CLP-Like packages for non-EPA clients.

2.0 RESPONSIBILITIES

2.1 It is the responsibility of the Inorganic Data Clerk to designate samples for a Sample Delivery Group (SDG) for EPA samples received at the laboratory. The Inorganic Data Clerk also has the responsibilities of SDG Cover Sheet maintenance and delivery, maintaining complete SDG File (CSF), data package assembly and pagination.

2.2 The responsibility for designating samples for a SDG for non-EPA samples belongs to the Project Officer. They also have the responsibility of notifying the laboratory that a CLP-like data package is required for the SDG.

2.3 The Inorganic Program Manager or his designee is responsible for receiving all data packages for correctness and completeness.

2.4 The QA/QC officer is responsible for receiving 10% of all non-EPA data packages for completeness.

2.5 The Sample Custodian is responsible for filling out the DC form or Cooler Receipt/Sample Login Sheet.

3.0 EQUIPMENT

3.1 Software

3.1.1 Ward Scientific, EDR/CLP Report Program, Version 3.19.

3.2 Hardware

3.2.1 PC-486 or 586 Intel based processors.

3.2.2 HP LaserJet Printer

3.2.3 Numbering Machine

3.2.4 Copier

4.0 PRECAUTIONS. Failure to comply with this SOP could result in liquidated damage penalties.

5.0 PROCEDURE

5.1 Required Forms

5.1.1 Figures 1 through 18 show the required forms to be included in a data package for EPA. Forms for CLP-like packages are identical with the exception that references to U.S.E.P.A. and SOW numbers are removed from the forms. As can be seen, six pieces of information are common to the header sections of each data reporting form.

5.1.1.1 Lab Name: Southwest Laboratory of Oklahoma, Inc.

5.1.1.2 Contract: The number of the EPA contract under which the analyses is performed. For CLP-like packages, the client name is placed here.

5.1.1.3 Lab Code: SWOK

5.1.1.4 Case Number: The EPA assigned case number associated with the samples, and reported on the Traffic Report.

5.1.1.5 SAS Number: The EPA assigned case number for analyses performed under Special Analytical Services.

5.1.1.6 SDG Number: Sample Delivery Group number.

5.1.2 Cover Page. This form is used to list client sample numbers in alphanumeric order analyzed within the Sample Delivery Group. It also cross-references the sample number with the laboratory ID. The information required to fill out this form is:

5.1.2.1 Header Information discussed in Section 5.1.1

5.1.2.2 The "SOW No." indicates the Statement of Work (SOW) version under which analyses in the data package have been performed.

5.1.2.3 Enter the client Sample Number, including the duplicates and spikes of every sample analyzed within the SDG. Spikes must contain a "S" suffix and duplicates a "D" suffix. List the samples in ascending alphanumeric order.

5.1.2.4 Enter the Lab Sample ID for each client Sample Number up to ten spaces may be used for the Lab Sample ID.

5.1.2.5 Enter "Yes" or "No" in answer to each of the two questions concerning ICP corrections. The third question must be answered with a "Yes" or "No", if the answer to the second question is "Yes". And if the answer to the second question is "No", then the third question should be left blank.

5.1.2.6 Under "Comments" enter any statements relevant to the analyses performed under the SDG as a whole.

5.1.2.7 Each cover page must be signed and dated by the Laboratory Manager or the Manager's assistant.

5.1.3 Inorganic Analysis Data Sheet (Form 1). This form is used to tabulate and report sample analysis results for target analytes.

5.1.3.1 Header information is discussed in Section 5.1.1.

5.1.3.2 Client Sample Number is entered into the triple-space box in the upper right hand corner and entered on the middle line.

5.1.3.3 For "Matrix" enter "Soil" for soil/sediment samples and enter "Water" for water samples.

5.1.3.4 For "Level" enter the determination of concentration level, "Low" or "Med".

5.1.3.5 "Date Received" is the date of sample receipt at the laboratory.

5.1.3.6 The results for the "% solids" will be entered here to one decimal place. If the percent solids is not required because the sample is fully aqueous or less than 1% solids a "0" will be entered.

5.1.3.7 Under "Concentration Units" enter UG/L for water or MG/KG for soils. Entering MG/KG means "mg/kg dry weight" on this form.

5.1.3.8 Under the column labeled "concentration" enter for each analyte the value of the result if the concentration is greater than or equal to the Instrument or Method Detection Limit. If the concentration is less than the Instrument or Method Detection Limit enter the Instrument or Method Detection Limit for the analyte corrected for any dilutions. Analytical results must be reported to two significant figures if the result values less than 10, to three significant figures if the result is greater than or equal to 10.

5.1.3.9 Under "C" (Concentration) enter a "B" if the reported value, is less than the Contract Required Detection Limit (CRDL) or Practical Quantitation Limit (PQL) but greater than the Instrument or Method Detection Limit. If the analyte was analyzed for but not detected, then a "U" must be entered. If the reported value is greater than or equal to the CRDL or PQL, then this column should be left blank.

5.1.3.10 The "Q" qualifier column has specific entries, which follow below.

E = The reported value is estimated because of the presence of interference. An explanatory note is included in the comments in the Case Narrative if the problem applies to all samples or on the specific Form I if it is an isolated problem.

M = Duplicate injection precision not met.

N = Spiked sample recovery not within control limits.

S = The reported value was determined by the Method of Standard Additions (MSA).

W = Post-digestion spike for Furnace AA analysis is out of control limits (85-115%), while sample absorbance is less than 50% of spike absorbance.

* = Duplicate analysis not within control limits.

+ = Correlation coefficient for the MSA is less than 0.995.

5.1.3.11 For the "M" (method) field enter either:

"P" for ICP

"A" for Flame AA

"AS" for Semi-Automated Spectrophotometric Cyanide

"AV" for Automated Cold Vapor AA

"F" for Furnace AA

"NR" if this analyte is not required to be analyzed.

5.1.3.12 A brief physical description of the sample, both before and after digestion, must be reported. For water samples, report color, texture and artifacts. If artifacts are present, then enter "Yes" in the artifact field and describe the artifacts in the Comments field. If artifacts are not present, then leave this field blank.

5.1.4 Initial and Continuing Calibration Verification (Form 2A). This form is used to report analyte recoveries from calibrated solutions.

5.1.4.1 Complete the header information as discussed in Section 5.1.1.

5.1.4.2 Enter both the sources for the Initial and continuing calibration Standards. Maximum of 12 spaces is allowed. Enter "EPA-LV" to indicate solutions received from EPA EMSL Las Vegas. If other sources were used, enter sufficient information in the available spaces to identify the manufacturer and the solution used.

5.1.4.3 Under "Initial Calibration True" enter the value in ug/l (to one decimal place of the concentration of each analyte in the Initial Calibration Verification Solution.

5.1.4.4 Under "Initial Calibration Found" enter the value in ug/l, (to two decimal places) of the concentration of each analyte measured in the Initial calibration Verification Solution.

5.1.4.5 Under "Initial Calibration % R" enter the percent recovery (to one decimal place) computed by the following equation:

$$\%R = \frac{\text{Found (ICV)}}{\text{True (ICV)}} \times 100$$

Where:

True (ICV) = True concentration of the analyte in the Initial Calibration Verification Solution

Found (ICV)= The Found Concentration of the analyte in the Initial Calibration Verification Solution.

5.1.4.6 Under "Continuing Calibration True" enter the value in ug/l (to one decimal place) of the concentration of each analyte in the Continuing Calibration Verification Solution.

5.1.4.7 Under "Continuing Calibration Found" enter the value in ug/l (to two decimal places) of the concentration of each analyte measured in the Continuing Calibration Verification Solution.

5.1.4.8 Under "Continuing Calibration % R", enter the percent Recovery (to one decimal place) computed by the above equation.

5.1.4.9 Under "M" enter the method used or "NR" if the analyte was not required.

5.1.5 CRDL Standard for AA and ICP (Form 2B). This form is used to report analyte recoveries from analyses of the CRDL standards for AA, ICP, and CV.

5.1.5.1 Complete the header information as discussed in Section 5.1.1.

5.1.5.2 Enter the AA CRDL or PQL Standard Source and the ICP CRDL or PQL Standard Source.

5.1.5.3 Under "CRDL" Standard for AA True" and "CRDL Standard for ICP True" enter the value in ug/l (to one decimal place) of the concentration of each analyte in the CRDL Standard Solution.

5.1.5.4 Under "CRDL Standard for AA Found" enter the value in the ug/l (to two decimal places) of the concentration of the analyte measured in the CRDL Standard Solution.

5.1.5.5 Under "CRDL Standard for AA % R" enter the percent Recovery to one decimal place.

5.1.5.6 Under "CRDL" Standard for ICP Initial Found" enter the value in ug/l (to two decimal places) of the concentration of each element measured in the CRDL Standard Solution analyzed at the beginning of the run.

5.1.5.7 Under "CRDL Standard for ICP Final Found" enter the value in ug/l (to two decimal places) of the concentration of each element measured in the CRDL standard Solution analyzed at the end of the run.

5.1.5.8 Under "CRDL Standard for ICP Initial % R" and "Final % R" enter the percent Recoveries to one decimal place.

5.1.6 Blanks (Form 3). This form is used to report concentrations found in the Initial Calibration Blank, Continuing Calibration Blanks, and the Preparation Blank.

5.1.6.1 Complete the header information as discussed in Section 5.1.1.

5.1.6.2 Enter "Soil" or "Water" as the matrix of the Preparation Blank.

5.1.6.3 Enter the appropriate concentration units for the Preparation Blank, ug/l or mg/kg.

5.1.6.4 Under "Initial Calibration Blank" if the absolute value of the analyte concentration is greater than the IDL or MDL, enter that value. Enter the IDL or MDL if the absolute value of the analyte is less than the IDL or MDL.

5.1.6.5 Enter the appropriate qualifier as discussed in Section 5.1.3.9 in the "C" qualifier column.

5.1.6.6 Under "Continuing Calibration Blank" enter the concentration in ug/l (to one decimal place) of the Continuing Calibration Blanks using the same criteria as the Initial Calibration Blank.

5.1.6.7 Enter the appropriate qualifier as discussed in Section 5.1.3.9 in the "C" qualifier columns.

5.1.6.8 Under "Preparation Blank" enter the concentration in ug/l (to one decimal place) for a water blank or in mg/kg (up to three decimal places) for a soil blank. Enter the concentration of each analyte measured above the IDL or MDL or below the negative value of the IDL or MDL.

5.1.6.9 Under the "C" qualifier field enter "B" if the absolute value of the analyte concentration is less than the CRDL or PQL but greater than or equal to the IDL or MDL. Enter "U" if the absolute value of the analyte in the blank is less than the IDL or MDL.

5.1.6.10 Under "M" enter the method used.

5.1.7 ICP Interference Check Sample (Form 4). This form is used to report Interference Check Sample (ICS) results for each ICP instrument used in Sample Delivery Group.

5.1.7.1 Complete the header information as discussed in Section 5.1.1.

5.1.7.2 For "ICP ID Number" enter TJA61E to identify the Thermal-Jarrell Ash ICP. If additional ICP's are added in the future they will be given their own unique ID.

- TJA ET1 for Thermal-Jarrell Ash Trace 1
- TJA ET2 for Thermal-Jarrell Ash Trace 2

5.1.7.3 For "ICS Source" enter the Standard Source.

5.1.7.4 Under "True Sol A" enter the true concentration in ug/l (to the nearest whole number) of each analyte present in Solution A.

5.1.7.5 Under "True Sol. AB" enter the true concentration in ug/l (to the nearest whole number) of each analyte present in Solution AB.

5.1.7.6 Under "Initial Found Sol. A." and "Final Found Sol. A", enter the concentration ug/l (to the nearest whole number) of each analyte found in the initial and final analysis of Solution A respectively.

5.1.7.7 Under "Initial Found So. AB" and "Final Found So. AB" enter the concentration in ug/l (to one decimal place) of each analyte found in the initial and final analysis of solution AB, respectively.

5.1.7.8 Under "Initial found % R" and "Final Found % R" enter the respective percent recoveries to one decimal place.

5.1.8 Spike Sample Recovery (Form 5A). This form is used to report the results from the pre-digested matrix spike.

5.1.8.1 Complete the header information as discussed in section 5.1.1.

5.1.8.2 Indicate the appropriate matrix, level and concentration units as those used for the sample on Form 1.

5.1.8.3 In the "EPA Sample No." Box, enter the client sample number (11 places maximum) of the sample from which the spike results on this form were obtained.

5.1.8.4 Under "Control Limits % R" enter "75-125" if the spike added value was greater than or equal to one fourth of the sample result.

5.1.8.5 Under "Spiked Sample Result (SSR)" enter the measured value (up to four decimal places), in appropriate units, for each analyte in the matrix spike sample. Enter any appropriate qualifier to the "C" qualifier column immediately following the "Spiked Sample Result (SSR)" column.

5.1.8.6 Under "Sample Result (SR)" enter the measured value (up to four decimal places) for each required analyte in the sample n which the matrix spike was performed. Enter any appropriate qualifier to the "C" qualifier column immediately following the "Sample Result (SR)".

5.1.8.7 Under "Spike Added (SA)" enter the value (to one decimal place) for the concentration of each analyte added to the sample.

5.1.8.8 Under "% R" enter the percent recovery (to one decimal place) computed by the following formula:

$$\% R = \frac{(SSR - SR)}{SA} \times 100$$

The % R must be reported whether it is negative, positive or zero. A value of zero must be used for calculating % R for the SSR or SR if the analyte value is less than the IDL or MDL.

5.1.8.9 The same concentration units must be used for spiked sample results, non-spiked results, and spike added sample results.

5.1.8.10 Under "Q" enter "N" if the Spike recovery is out of the control limits (75-125) and the Spike added (SA) is greater than or equal to one-fourth of the Sample Result (SR).

5.1.8.11 Under "M", enter the method used or enter "NR" if the analyte is not required in the spike.

5.1.8.12 If different samples are used for spike sample analysis of different analytes, a separate Form must be submitted for each sample used.

5.1.9 Post Digest Sample Recovery (Form 5B). This form is used to report results for the post-digest spike recovery, which is based upon the addition of a known quantity of analyte to an aliquot of the digested sample.

5.1.9.1 Complete the header information as discussed in Section 5.1.1.

5.1.9.2 In the "EPA Sample No." Box enter the client Sample number (11 spaces maximum) of the sample from which the spike results on this form were obtained.

5.1.9.3 The "Control Limits % R" and "Q" field are left blank.

5.1.9.4 Under "Spike Sample Result (SSR)" enter the measured value in ug/l (to two decimal places) for each analyte in the post-digestion spike. Enter any appropriate qualifier to the "C" qualifier column.

5.1.9.5 Under "Sample Result (SR)" enter the measured value in ug/l (to two decimal places) for the concentration of each analyte in the sample on which the spike was performed. Enter any appropriate qualifier to the "C" qualifier column.

5.1.9.6 Under "Spike Added (SA)" enter the value in ug/l (to one decimal place) for each analyte added to the sample.

5.1.9.7 Under "% R", enter the value (to one decimal point) of the percent recovery using the same equation as in Section 5.1.8.8. The % R must be reported, whether it is negative, positive or zero. A Value of zero is used for SSR or SR if the analyte value is less than the IDL or MDL.

5.1.9.8 Under "M", enter the method used or enter "NR" if the spike was not required.

5.1.10 Duplicates (Form 6). This form is used to report results of duplicate analyses. Duplicate analyses are required for all analytes including % solids.

5.1.10.1 Complete the header information as discussed in Section 5.1.1.

5.1.10.2 Indicate the appropriate matrix, level and concentration units.

5.1.10.3 In the "EPA Sample No." box, enter the client Sample Number (11 spaces maximum) of the sample used for duplicate analyses.

5.1.10.4 Under "Control Limits" enter the CRDL or PQL (in appropriate units) for the analyte if the sample or duplicate values were less than 5X CRDL or PQL. If not, leave the field empty.

5.1.10.5 Under "Sample (S)" enter the measured value (up to four decimal places) for the original sample. Enter an appropriate qualifier to the "C" qualifier column immediately after the "Sample (S)" column.

5.1.10.6 Under "Duplicate (D)" enter the measured value (up to four decimal places) for each analyte in the Duplicate Sample. Enter any appropriate qualifier to the "C" column immediately following the "Duplicate (D)" column.

5.1.10.7 Under "RPD" enter the absolute value (to one decimal place) of the relative percent difference for all analytes detected above the IDL or MDL in either the sample or the duplicate, using the following formula:

$$RPD = \frac{S - D}{(S + D)/2} \times 100$$

A value of zero must be used or S or D if the concentration is less than the IDL or MDL in either one. If the concentration is less than the IDL or MDL in both S and D, leave the RPD field empty.

5.1.10.8 Under "Q" enter "*" if the duplicate analysis for the analyte is out of control. If both the sample and duplicate values are greater than 5 X CRDL or PQL, then the RPD must be less than or equal to 20% to be in control. If either sample or duplicate values are less than or equal to 5 X the CRDL or PQL, then the absolute difference between the two values must be less than the CRDL or PQL to be in control. If both the values are below the CRDL or PQL, then no control limit is applicable.

5.1.10.9 Under "M" enter the method used.

5.1.11 Laboratory Control Sample (Form 7)

5.1.11.1 This form is used to report results for the solid and aqueous Laboratory Control Samples.

5.1.11.2 Complete the header information as discussed in Section 5.1.1.

5.1.11.3 For the Solid LCS Source enter the Standard Source.

5.1.11.4 Under "Aqueous True" enter the value in ug/l (to one decimal place) of the concentration of each analyte in the Aqueous LCS Standard Source.

5.1.11.5 Under "Aqueous Found" enter the measured concentration in ug/l (to two decimal places) of each analyte.

5.1.11.6 Under "Aqueous % R" enter the value of the percent recovery.

5.1.11.7 Under "Solid True" enter the value in mg/kg (to one decimal place) of the concentration of each analyte in the Solid LCS Source.

5.1.11.8 Under "Solid Found" enter the measured value in mg/kg (to one decimal place) of each analyte.

5.1.11.9 Under "C" enter "B" or "U" or leave empty, to describe the found value of the solid LCS as explained in Section 5.1.3.9.

5.1.11.10 Under "Limits" enter the lower limit in mg/kg (to one decimal place) in the left column, and the upper limit in mg/kg (to one decimal place) in the right hand column.

5.1.11.11 Under "Solid % R" enter the value of the percent recovery (to one decimal place).

5.1.12 Standard Addition Results (Form 9)

5.1.12.1 This form is used to report the results of samples analyzed using the Method of Standard Additions (MSA) for furnace AA analysis.

5.1.12.2 Complete the header information as discussed in Section 5.1.1.

5.1.12.3 Under "EPA Sample No." enter the client Sample Numbers (11 spaces maximum) of all analytical samples analyzed using MSA. This includes reruns by MSA (if the first MSA was out of control).

5.1.12.4 Under "AN" enter the chemical symbol for each analyte for which MSA was required. The analyte must be in Alphanumeric order per analyte.

5.1.12.5 Under "Dil" enter the dilution factor used to obtain the ABS values on this form. Enter "1" if no dilution was necessary.

5.1.12.6 Under "O ADD ABS" enter the measured value in absorbance units or ug/l for the analyte before addition is performed.

5.1.12.7 Under "1 ADD CON" enter the concentration of the first addition to the sample analyzed by MSA.

5.1.12.8 Under "1 ADD ABS", enter the measured value of the sample solution spiked with the first addition.

5.1.12.9 Under "2 ADD CON", enter the concentration of the second addition to the sample analyzed by MSA.

5.1.12.10 Under "2 ADD ABS", enter the measured value of the sample solution spiked with the second addition.

5.1.12.11 Under "3 ADD", enter the concentration of the third addition to the sample analyzed by MSA.

5.1.12.12 Under "3 ADD ABS" enter the measured value of the sample solution spiked with the third addition.

5.1.12.13 Under "Final Conc.", enter the final analyte concentration in ug/l (to one decimal place) in the sample as determined by MSA.

5.1.12.14 Under "r" enter the correlation coefficient (up to four decimal places) that is obtained from the least squares regression line.

5.1.12.15 Under "Q" enter "+" if r is less than 0.995.

5.1.13 ICP Serial Dilution (Form 9)

5.1.13.1 This form is used to report results for ICP serial dilution.

5.1.13.2 Complete the header information as discussed in Section 5.1.1.

5.1.13.3 In the "EPA Sample No." Box, enter the client Sample Number of the sample for which serial dilution analysis results on this form were obtained.

5.1.13.4 Under "Initial Sample Result (I)", enter the measured value in ug/l (up to two decimal places) for each analyte in the undiluted sample on which a 5 X dilution was performed. The initial Sample Concentration for analyte does not have to equal the value for that analyte reported on Form I for that sample. It is the value of the analyte concentration (uncorrected for dilution) that is within the linear range of the instrument.

5.1.13.5 Enter any appropriate qualifier to the "C" qualifier column immediately following the "Initial Sample Results (I)" column.

5.1.13.6 Under "Serial Dilution Result (S)", enter the measured concentration value in ug/l (up to two decimal places) for each required analyte in the diluted sample. The serial Dilution Results (S) is obtained by multiplying by five the instrument measured value (in ug/l) of the serially diluted sample.

5.1.13.7 Under "% Difference", enter the absolute value (to one decimal place) of the percent difference between the original sample and the diluted sample (adjusted for dilution) according to the following formula:

$$\% \text{ Difference} = \frac{1 = S}{I} \text{ X } 100$$

If the analyte concentration in (I) is less than the IDL or MDL, then leave the "% Difference" field empty.

5.1.13.8 Under "Q" enter "E" if the % Difference is greater than 10% and the original sample concentration (reported on Form I) is greater than 50 X the IDL or MDL.

5.1.13.9 Under "M" enter the method of analysis for each analyte. Enter "NR" for each analyte on which serial dilution was not performed because the concentration of analyte, in the original sample was less than 50 X the IDL or MDL. Any entry under "M" other than "P" or "NR" indicates that the serial dilution was not required because the analyte was not analyzed by ICP and thus no values must appear in the (I), (S) or % Difference columns.

5.1.14 Instrument Detection Limits (Quarterly) (Form 10)

5.1.14.1 This form documents the Instrument Detection Limits or Method Detection Limits for private clients, for each instrument that the laboratory used to obtain data for the Sample Delivery Group.

5.1.14.2 Complete the header information as discussed in Section 5.1.1.

5.1.14.3 Enter the date on which the IDL or MDL values were obtained (or became effective). The date must not exceed or precede the analysis dates in the SDG data package by more than three months for EPA.

5.1.14.4 Enter ICP ID number, Flame AA ID number, and Furnace AA ID number. These ID numbers are used to uniquely identify each instrument that the laboratory uses to do analytical work.

5.1.14.5 Under "Wavelength" enter the wavelength in nanometers (to two decimal places) for each analyte for which an IDL or MDL has been established and is listed in an IDL column.

5.1.14.6 Under "Background" enter the type of background correction used to obtain Furnace AA data: Enter "BS" for Smith Hieftje, "BD" for Deuterium ARC, or "BZ" for Zeeman Background correction.

5.1.14.7 Under "IDL" enter the IDL or MDL in ug/l (up to one decimal place) as determined by the laboratory for each analyte analyzed by the instrument for which the ID number is listed on the form.

5.1.14.8 Under "M" enter the method of analysis used to determine the IDL or MDL for each wavelength used.

5.1.15 ICP Interelement Correction Factors (Quarterly) (Form 11A & 11B)

5.1.15.1 This form documents for each ICP instrument the interelement correction factors applied to obtain data for the Sample Delivery Group. Although the correction factors are determined annually, a copy of the quarterly interelement correction factors must be included with each SDG data package.

5.1.15.2 Complete the header information as discussed in Section 5.1.1.

5.1.15.3 Enter ICP, either TJA61E, TJA ET1, or TJA ET2 in the ICP ID number.

5.1.15.4 Report the date on which the correction factors were determined for use. This date must not exceed the ICP analysis dates in the SDG data package or precede them by more than twelve calendar months.

5.1.15.5 Under "Wavelength" list the wavelength in nanometers (to two decimal places) used for each analyte analyzed by ICP.

5.1.15.6 Under "Al", "Ca", "Fe", "Mg", enter the correction factors (negative or positive, up to seven decimal places) for each corrected analyte analyzed by ICP. If correction factors for another analyte are applied, use the empty column and list the analyte's chemical symbol in the blank two-space header field provided for that column.

5.1.15.7 If additional correction factors for analytes are applied use Form 12B. Complete this form as for Form 12A by listing the chemical symbol for additional analyses in the heading of the empty columns in the two-space fields provided.

5.1.16 ICP Linear Ranges (Quarterly) (Form 12)

5.1.16.1 This form documents the quarterly linear range analysis for each ICP instrument that the laboratory used to obtain data for the SDG.

5.1.16.2 Complete the header information as discussed in Section 5.1.1.

5.1.16.3 Enter TJA61E, TJA ET1, or TJA ET2 for the ICP ID Number.

5.1.16.4 Report the date on which the linear ranges were determined. This date must not exceed the dates of analysis by ICP in the SDG data package and must not precede the analysis dates by more than three calendar months.

5.1.16.5 Under "Integ. Time (Sec.)", enter the integration time (in seconds to two decimal places) used for each measurement taken from the ICP instrument.

5.1.16.6 Under "Concentration" enter the concentration (in ug/l) that is the upper limit of the ICP instrument linear range. Any measurement in the SDG data package at or below this concentration is within the linear range. Any measurement above it, is out of linear range, and thus, is an estimated value and must be diluted into the linear range.

5.1.16.7 Under "M", enter the "NA" for analytes analyzed by methods other than ICP. No entries are required under "M" for analytes analyzed by ICP.

5.1.17 Preparation Log (Form 13)

5.1.17.1 This Form is used to report the preparation run log.

5.1.17.2 Complete the header information as discussed in Section 5.1.1.

5.1.17.3 For "Method", enter the method of analysis (two characters maximum) for which the preparations listed on the Form were made. Use appropriate method codes as specified in Section 5.1.3.

5.1.17.4 Under "EPA Sample No.", enter the client Sample number of each sample in the SDG, and of all other preparations such as duplicates, matrix spikes, LCS's, PB's, and repreparations. All client Sample Numbers shall be listed in ascending alphanumeric order, continuing to the next Form 13, if applicable.

5.1.17.5 Under "Preparation Date", enter the date (formatted MM/DD/YY) on which each sample was prepared for analysis by the method indicated in the header section of the Form.

5.1.17.6 Under "Weight", enter the wet weight (in grams, to two decimal places) of each soil sample prepared for analysis by the method indicated in the header section of the Form. If the sample matrix, is water, then leave the field empty.

5.1.17.7 Under "Volume", enter the final volume (in mL, to the nearest whole number) of the preparation of each sample prepared for analysis by the method indicated in the header section of the Form. This field shall have a value for each sample listed.

5.1.18 Analysis Run Log (Form 14)

5.1.18.1 This Form is used to report the sample analysis run log.

5.1.18.2 Complete the header information as discussed in Section 5.1.1.

5.1.18.3 For "Instrument ID Number", enter the instrument ID number (12 spaces maximum) which shall be an identifier designated by the laboratory to uniquely identify each instrument used to produce data which are required to be reported in the SDG deliverable. If more than one instrument is used, submit additional Forms 14 as appropriate.

5.1.18.4 For "Method", enter the method code (two characters maximum) according to the specifications in Section 5.1.3.

5.1.18.5 For "Start Date", enter the date (formatted MM/DD/YY) on which the analysis run was started.

5.1.18.6 For "End Date", enter the date (formatted MM/DD/YY) on which the analysis run was ended.

5.1.18.7 Under "EPA sample No.", enter the client sample number of each analysis, including all QC operations applicable to the SDG. All client Sample Numbers shall be listed in increasing temporal (date and time) order of analysis, continuing to the next Form 14 for the instrument run if applicable. The analysis date and time of other analyses not associated with the SDG, but analyzed by the instrument in the reported analytical run, shall be reported. Those analyses shall be identified with the client Sample Number of "ZZZZZZ".

5.1.18.8 Under "D/F", enter the dilution factor (to two decimal places) by which the final digestate or distillate needed to be diluted for each analysis to be performed. The dilution factor does not include the dilution inherent in the preparation as specified by the preparation procedures.

The dilution factor is required for all entries on Form 14.

5.1.18.9 Under "Time", enter the time (in military format — HHMM) at which each analysis was performed. If an autosampler is used with equal analysis time and intervals between analyses, then only the start time of the run (the time of analysis of the first calibration standard). and end time of the run (the time of analysis of the final CCV or CCB, whichever is later), need to be reported.

5.1.18.10 Under "% R", enter the percent recovery (to one decimal place) for each furnace AA analytical spike analyzed. If the analytical spike was performed on more than one analyte, use additional Forms 14 as appropriate. Leave the "% R" field empty if the analysis reported is not for an analytical spike. % R shall be recorded even if the result is not used.

A % R value of "-9999.9" shall be entered for the analytical spike if either the sample or analytical results is greater than the calibration range of the instrument.

5.1.18.11 Under "Analytes", enter "X" in the column of the designated analyte to indicate that the analyte value was used from the reported analysis to report data in the SDG. Leave the column empty for each analyte if the analysis was not used to report the particular anlayte.

Entering "X" appropriately is very important. The "X" is used to link the samples with their related QC. It also links the dilution factor with the appropriate result reported on Forms 1-9. For each analyte result reported on any of the Forms 1-9, there shall be one, and only one, properly identified entry on Form 14 for which an "X" is entered in the column for the analyte.

5.2 Raw Data

5.2.1 For each reported value, all raw data from the instrument used to obtain the value and the QA/QC values reported must be included in the data package. All AA and ICP instruments must provide a legible hard copy of the direct real-time instrument readout. A hard copy of the direct instrument readout for cyanide must be included.

5.2.2 The order of raw data in the package is:

5.2.2.1 ICP

5.2.2.2 Flame AA

5.2.2.3 Furnace AA

5.2.2.4 Mercury

5.2.2.5 Cyanide

5.2.3 The raw data must be labeled with client Sample Numbers and be able to identify:

5.2.3.1 Calibration Standards: including source and prep date.

5.2.3.2 All QA/QC samples.

5.2.3.3 Diluted and undiluted samples and all weights, dilutions and volumes used to obtain the reported values.

5.2.3.4 Duplicates

5.2.3.5 Matrix spikes including standard solutions used, final spike concentrations, and volumes.

5.2.3.6 Instruments used, any instrument adjustments, any data corrections or other apparent anomalies on the measurement record, including all data voided or data not used to obtain reported values and a brief written explanation.

5.2.3.7 Furnace data should include client Sample Number, sample and analytical spike data, percent recovery, coefficient of variation, full MSA data, MSA correlation coefficient, slope and y intercept of linear fit, final sample concentration and type of background-correction used.

5.2.3.8 The time and date for initial and continuing calibration verifications and blanks, as well as interference check samples and linear range analysis.

5.2.4 The digestion and distillation logs must include the date, sample weights and volumes, sufficient information to unequivocally identify which QC samples correspond to each batch and an indication of the pH being <2 or >12 as applicable.

5.3 SDG File

5.3.1 As specified in the Delivery Schedule, one Complete SDG File (CSF) including the original Sample Data Package shall be delivered to the Region concurrently with delivery of a copy of the Sample Data Package to SMO (delivery to QATS is only required upon written request). The CSF shall contain all original documents where possible. No copies of original documents shall be placed in

the CSF unless the originals are bound in a logbook maintained by the laboratory. Private client information is contained in the clients' project file, which is discussed in SOP SWL-GA-132.

5.3.2 SDG Cover Sheets. This is sent prior to conclusion of complete SDG package and includes the following:

5.3.2.1 Lab name

5.3.2.2 Contract number

5.3.2.3 Sample Analysis Price — full sample price from contract

5.3.2.4 Case Number

5.3.2.5 List of EPA Sample Numbers of all samples in the SDG, identifying the first and last samples received, and their dates of receipt.

Note: When more than one sample is received in the first or last SDG shipment, the "first" sample received would be the sample with the lowest sample number (considering both alpha and numeric designations and shipment date); the "last" sample received would be the sample with the highest sample number (considering both alpha and numeric designations plus shipment date).

5.3.3 Communication. All original communications in shipping and receiving and original telephone logs that provide information on SDG.

5.3.4 Airbills. Original Airbill sent with samples.

5.3.5 Traffic Report. Original Traffic Reports marked Lab Copy for return to region are included at the back of the data package. The Traffic Reports are arranged in increasing EPA Sample Number order, considering both alpha and numeric designations and date received.

5.3.6 Sample Login Sheet. Original DC-1 form filled out by Sample Receiving.

5.3.7 Sample Tag Summary. Contains the following: (This is included only if tags are sent with samples.)

5.3.7.1 Lab Name

5.3.7.2 Contract Number

5.3.7.3 RAS Number

5.3.7.4 List of EPA Sample Numbers. One of each tag sent in alphanumeric order by received

5.3.8 Package Cover Sheet to be sent to SMO.

5.3.8.1 Lab code

5.3.8.2 Case Number

5.3.8.3 SDG Number

5.3.8.4 SAS Number (if applicable).

5.3.8.5 Contract Number

5.3.8.6 Place check next to correct SOW Number (Fig. 19).

5.3.8.7 Place check by the type of package sent (Fig.19).

5.3.9 Document Inventory Sheet. (Form DC-2 to be sent to Region)

5.3.9.1 This form is used to record the inventory of the complete SDG File (CSF) documents, which are sent to the Region.

5.3.9.2 Organize all EPA-CSF documents as described in Section IV. Assemble the documents in the order specified on Form DC-2 and Section 2, and stamp each page with the consecutive number. (Do not number Form DC-2). Inventory the CSF by reviewing the document numbers and recording page number ranges in the columns provided on Form DC-2. If there are no documents for a specific document type, enter a "NA" in the empty space.

5.3.9.3 Certain laboratory-specific documents related to the CSF may not fit into a clearly defined category. The laboratory should review Form DC-2 to determine if it is most appropriate to place them under Categories 29, 30, 31, or 32. Category 32 should be used if there is no appropriate previous category. These types of documents should be described or listed in the blanks under each appropriate category.

5.3.9.4 If it is necessary to insert new or inadvertently omitted documents prior to providing CSF's as first deliverables, the Contractor shall follow these steps:

- Number all documents to be inserted with the next sequential numbers and file the inserts in their logical position within the CSF (e.g., file document 1000 between documents 6 and 7).
- Identify where the inserts are filed in the CSF by recording the document numbers and their locations under the "Other Records" section of Form DC-2 (e.g., document 1000 is filed between 6 and 7).

6.0 RECORDS

6.1 Inorganic Data Package

6.2 Traffic Report

6.3 DC-1 Form

6.4 Cooler Receipt/Sample Login Form

6.5 SDG Cover Sheets

6.6 Client Communication Logs

7.0 REFERENCES.

USEPA Contract Laboratory Program Statement of Work for Inorganic Analysis, multi-media, multi-concentration. Document Number ILM04.1.

8.0 ATTACHMENTS

8.1 Figure 1: Reference 5.1.2 "Cover Page — Inorganic Analysis Data Package"

8.2 Figure 2: Reference 5.1.3 "Form 1 — Inorganic Analysis Data Sheet"

8.3 Figure 3: Reference 5.1.4 "Form 2A — Initial & Continuing Calibration Verification"

8.4 Figure 4: Reference 5.1.5 "Form 2B — CRDL Standard for AA and ICP"

8.5 Figure 5: Reference 5.1.6 "Form 3 — Blanks"

8.6 Figure 6: Reference 5.1.7 "Form 4 — ICP Interference Check Sample"

8.7 Figure 7: Reference 5.1.8 "Form 5A — Spike Sample Recovery"

8.8 Figure 8: Reference 5.1.9 "Form 5B — Post Digest Spike Sample Recovery"

8.9 Figure 9: Reference 5.1.10 "Form 6 — Duplicates"

8.10 Figure 10: Reference 5.1.11 "Form 7 — Laboratory Control Sample"

8.11 Figure 11: Reference 5.1.12 "Form 8 — Standard Addition Results"

8.12 Figure 12: Reference 5.1.13 "Form 9 — ICP Serial Dilution"

8.13 Figure 13: Reference 5.1.14 "Form 10 — Instrument Detection Limits"

8.14 Figure 14: Reference 5.1.15 "Form 11A — ICP Interelement Correction Factors"

8.15 Figure 15: Reference 5.1.15 "Form 11B — ICP Interelement Correction Factors"

8.16 Figure 16: Reference 5.1.16 "Form 12 — ICP Linear Ranges"

8.17 Figure 17: Reference 5.1.17 "Form 13 — Preparation Log"

8.18 Figure 18 — Reference 5.1.18 "Form 14 — Analysis Run Log"

8.19 Figure 19 — Reference 5.3.8 "SMO Package Cover Sheet"

8.20 Figure 20 — Reference 5.3.9 "DC-2 Form"

9.0 DEFINITIONS

9.1 Terminology

Case:.....A finite, usually predetermined number of samples collected over a given time period from a particular site. A case consists of one or more sample deliver groups.

Sample Delivery Group: ..A unit within a sample case that is used to identify a group of samples for delivery. An SDG is a group of 20 or fewer samples. Samples may be assigned to Sample Delivery Groups by matrix (i.e., all soils in one SDG, all waters in another).

Sample Number: .....A unique identification number designated by the client for each sample. The EPA Sample Number appears on the Sample Traffic Report, which documents information on that sample. Private client sample numbers appear on the chain-of-custody.

Traffic Report: .....On EPA sample identification form filled out by the sampler, which accompanies the sample during shipment to the laboratory and which is used for documenting sample condition and receipt by the laboratory.

9.2 Acronyms

EPA:.....United States Environmental Protection Agency

CLP:.....Contract Laboratory Program

SDG: .....Sample Delivery Group

CSF: .....Complete SDG File

QA/QC:.....Quality Assurance/Quality Control

SAS:.....Special Analytical Services

IDL:.....Instrument Detection Limit

MDL:.....Method Detection Limit

CRDL:.....Contract Required Detection Limit

PQL:.....Practical Quantitation Limit

MSA:.....Method of Standard Additions

ICP: .....Inductively Coupled Argon Plasma

AA:.....Atomic Absorption

ICV:.....Initial Calibration Verification

ICB:.....Initial Calibration Blank

ICS: .....Interference Check Sample

LCS: .....Laboratory Control Sample

SMO:.....Sample Management Office

RAS:.....Routine Analytical Services

SOW:.....Statement of Work

U.S. EPA - CLP

COVER PAGE - INORGANIC ANALYSES DATA PACKAGE

Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

SOW No.: _____

EPA Sample No.	Lab Sample ID.

Were ICP interelement corrections applied? Yes/No ___

Were ICP background corrections applied? Yes/No ___
If yes-were raw data generated before application of background corrections? Yes/No ___

Comments:

I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed above. Release of the data contained in this hardcopy data package and in the computer-readable data submitted on diskette has been authorized by the Laboratory Manager or the Manager's designee, as verified by the following signature.

Signature: _____ Name: _____

Date: _____ Title: _____

COVER PAGE - IN

Fig.1 Cover Page (Inorganic Analyses Data Package)

U.S. EPA - CLP

1
INORGANIC ANALYSIS DATA SHEET

EPA SAMPLE :

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Matrix (soil/water): _____ Lab Sample ID: _____

Level (low/med): _____ Date Received: _____

% Solids: _____

Concentration Units (ug/L or mg/kg dry weight): _____

CAS No.	Analyte	Concentration	C	Q	M
7429-90-5	Aluminum				
7440-36-0	Antimony				
7440-38-2	Arsenic				
7440-39-3	Barium				
7440-41-7	Beryllium				
7440-43-9	Cadmium				
7440-70-2	Calcium				
7440-47-3	Chromium				
7440-48-4	Cobalt				
7440-50-8	Copper				
7439-89-6	Iron				
7439-92-1	Lead				
7439-95-4	Magnesium				
7439-96-5	Manganese				
7439-97-6	Mercury				
7440-02-0	Nickel				
7440-09-7	Potassium				
7782-49-2	Selenium				
7440-22-4	Silver				
7440-23-5	Sodium				
7440-28-0	Thallium				
7440-42-2	Vanadium				
7440-66-6	Zinc				
	Cyanide				

Color Before: _____ Clarity Before: _____ Texture: _____

Color After: _____ Clarity After: _____ Artifacts: _____

Comments:

Fig. 2 Form I (Inorganic Analysis Data Sheet)

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2A
INITIAL AND CONTINUING CALIBRATION VERIFICATION

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Initial Calibration Source: _____

Continuing Calibration Source: _____

Concentration Units: ug/L

Analyte	Initial Calibration True	Found	%R(1)	Continuing Calibration True	Found	%R(1)	Found	%R(1)	M
Aluminum									
Antimony									
Arsenic									
Barium									
Beryllium									
Cadmium									
Calcium									
Chromium									
Cobalt									
Copper									
Iron									
Lead									
Magnesium									
Manganese									
Mercury									
Nickel									
Potassium									
Selenium									
Silver									
Sodium									
Thallium									
Vanadium									
Zinc									
Cyanide									

(1) Control Limits: Mercury 80-120; Other Metals 90-110; Cyanide 85-115

FORM II (PART 1) - IN

Fig. 3 Form II – Part 1 (Initial & Continuing Calibration Verification)

U.S. EPA - CLP

2B
CRDL STANDARD FOR AA AND ICP

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

AA CRDL Standard Source: _____

ICP CRDL Standard Source: _____

Concentration Units: ug/L

Analyte	CRDL Standard for AA True	Found	%R	CRDL Standard for ICP Initial True	Found	%R	Final Found	%R
Aluminum								
Antimony								
Arsenic								
Barium								
Beryllium								
Cadmium								
Calcium								
Chromium								
Cobalt								
Copper								
Iron								
Lead								
Magnesium								
Manganese								
Mercury								
Nickel								
Potassium								
Selenium								
Silver								
Sodium								
Thallium								
Vanadium								
Zinc								

Control Limits: no limits have been established by EPA at this time

FORM II (PART 2) - IN

Fig.4 Form II – Part 2 (CRDL Standard for AA and ICP)

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3
BLANKS

Lab Name: _____ Contract: _____

Lab Code: ____ Case No.: ____ SAS No.: ____ SDG No.: ____

Preparation Blank Matrix (soil/water): ____

Preparation Blank Concentration Units (ug/L or ug/kg): ____

Analyte	Initial Calib. Blank (ug/L)	C	Continuing Calibration Blank (ug/L) 1	C	2	C	3	C	Prepa-ration Blank	C	M
Aluminum											
Antimony											
Arsenic											
Barium											
Beryllium											
Cadmium											
Calcium											
Chromium											
Cobalt											
Copper											
Iron											
Lead											
Magnesium											
Manganese											
Mercury											
Nickel											
Potassium											
Selenium											
Silver											
Sodium											
Thallium											
Vanadium											
Zinc											
Cyanide											

FORM III - IN

Fig. 5 Form III (Blanks)

U.S. EPA - CLP

4
ICP INTERFERENCE CHECK SAMPLE

Lab Name: _____ Contract: _____

Lab Code: ____ Case No.: ____ SAS No.: ____ SDG No.: ____

ICP ID Number: _____ ICS Source: _____

Concentration Units: ug/L

Analyte	True Sol. A	True Sol. AB	Initial Found Sol. A	Initial Found Sol. AB	Initial Found %R	Final Found Sol. A	Final Found Sol. AB	Final Found %R
Aluminum								
Antimony								
Arsenic								
Barium								
Beryllium								
Cadmium								
Calcium								
Chromium								
Cobalt								
Copper								
Iron								
Lead								
Magnesium								
Manganese								
Mercury								
Nickel								
Potassium								
Selenium								
Silver								
Sodium								
Thallium								
Vanadium								
Zinc								

FORM IV - IN

Fig. 6 Form IV (ICP Interference Check Sample)

U.S. EPA - CLP

5A
SPIKE SAMPLE RECOVERY

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: ____ Case No.: ____ SAS No.: ____ SDG No.: ____

Matrix (soil/water): ____ Level (low/med): ____

% Solids for Sample: ____

Concentration Units (ug/L or mg/kg dry weight): ____

Analyte	Control Limit %R	Spiked Sample Result (SSR)	C	Sample Result (SR)	C	Spike Added (SA)	%R	Q	M
Aluminum									
Antimony									
Arsenic									
Barium									
Beryllium									
Cadmium									
Calcium									
Chromium									
Cobalt									
Copper									
Iron									
Lead									
Magnesium									
Manganese									
Mercury									
Nickel									
Potassium									
Selenium									
Silver									
Sodium									
Thallium									
Vanadium									
Zinc									
Cyanide									

Comments:

FORM V (PART 1) - IN

Fig. 7 Form V-Part 1 (Spike Sample Recovery)

U.S. EPA - CLP

5B
POST DIGEST SPIKE SAMPLE RECOVERY

EPA SAMPLE N

Lab Name: _____ Contract: _____

Lab Code: ____ Case No.: ____ SAS No.: ____ SDG No.: ____

Matrix (soil/water): ____ Level (low/med): ____

Concentration Units: ug/L

Analyte	Control Limit %R	Spiked Sample Result (SSR)	C	Sample Result (SR)	C	Spike Added (SA)	%R	Q	M
Aluminum									
Antimony									
Arsenic									
Barium									
Beryllium									
Cadmium									
Calcium									
Chromium									
Cobalt									
Copper									
Iron									
Lead									
Magnesium									
Manganese									
Mercury									
Nickel									
Potassium									
Selenium									
Silver									
Sodium									
Thallium									
Vanadium									
Zinc									
Cyanide									

Comments:

FORM V (PART 2) - IN

Fig. 8 Form V-Part 2 (Post Digest Spike Sample Recovery)

U.S. EPA - CLP

6
DUPLICATES

EPA SAMPLE NO.

Lab Name: ______ Contract: ______

Lab Code: ______ Case No.: ______ SAS No.: ______ SDG No.: ______

Matrix (soil/water): ______ Level (low/med): ______

% Solids for Sample: ______ % Solids for Duplicate: ______

Concentration Units (ug/L or mg/kg dry weight): ______

Analyte	Control Limit	Sample (S)	C	Duplicate (D)	C	RPD	Q	M
Aluminum								
Antimony								
Arsenic								
Barium								
Beryllium								
Cadmium								
Calcium								
Chromium								
Cobalt								
Copper								
Iron								
Lead								
Magnesium								
Manganese								
Mercury								
Nickel								
Potassium								
Selenium								
Silver								
Sodium								
Thallium								
Vanadium								
Zinc								
Cyanide								

FORM VI - IN

Fig. 9 Form VI (Duplicates)

U.S. EPA - CLP

7
LABORATORY CONTROL SAMPLE

Lab Name: ______ Contract: ______

Lab Code: ______ Case No.: ______ SAS No.: ______ SDG No.: ______

Solid LCS Source: ______

Aqueous LCS Source: ______

Analyte	Aqueous (ug/L) True	Found	%R	Solid (mg/kg) True	Found	C	Limits		%R
Aluminum									
Antimony									
Arsenic									
Barium									
Beryllium									
Cadmium									
Calcium									
Chromium									
Cobalt									
Copper									
Iron									
Lead									
Magnesium									
Manganese									
Mercury									
Nickel									
Potassium									
Selenium									
Silver									
Sodium									
Thallium									
Vanadium									
Zinc									
Cyanide									

FORM VII - IN

Fig. 10 Form VII (Laboratory Control Sample)

U.S. EPA - CLP

8
STANDARD ADDITION RESULTS

Lab Name: ______ Contract: ______

Lab Code: ______ Case No.: ______ SAS No.: ______ SDG No.: ______

Concentration Units: ug/L

EPA Sample No.	An	0 ADD ABS	1 ADD CON	ABS	2 ADD CON	ABS	3 ADD CON	ABS	Final Conc.	r	Q

FORM VIII - IN

Fig. 11 Form VIII (Standard Addition Results)

U.S. EPA - CLP

9
ICP SERIAL DILUTIONS

EPA SAMPLE NO.

Lab Name: ______ Contract: ______

Lab Code: ______ Case No.: ______ SAS No.: ______ SDG No.: ______

Matrix (soil/water): ______ Level (low/med): ______

Concentration Units: ug/L

Analyte	Initial Sample Result (I)	C	Serial Dilution Result (S)	C	% Difference	Q	M
Aluminum							
Antimony							
Arsenic							
Barium							
Beryllium							
Cadmium							
Calcium							
Chromium							
Cobalt							
Copper							
Iron							
Lead							
Magnesium							
Manganese							
Mercury							
Nickel							
Potassium							
Selenium							
Silver							
Sodium							
Thallium							
Vanadium							
Zinc							

FORM IX - IN

Fig. 12 Form IX (ICP Serial Dilutions)

U.S. EPA - CLP

10
INSTRUMENT DETECTION LIMITS (QUARTERLY)

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

ICP ID Number: _____ Date: _____

Flame AA ID Number: _____

Furnace AA ID Number: _____

Analyte	Wave-length (nm)	Back-ground	CRDL (ug/L)	IDL (ug/L)	M
Aluminum			200		
Antimony			60		
Arsenic			10		
Barium			200		
Beryllium			5		
Cadmium			5		
Calcium			5000		
Chromium			10		
Cobalt			50		
Copper			25		
Iron			100		
Lead			3		
Magnesium			5000		
Manganese			15		
Mercury			0.2		
Nickel			40		
Potassium			5000		
Selenium			5		
Silver			10		
Sodium			5000		
Thallium			10		
Vanadium			50		
Zinc			20		
Cyanide			10		

Comments:

FORM X - IN

Fig. 13 Form X (Instrument Detection Limits, Quarterly)

U.S. EPA - CLP

11A
ICP INTERELEMENT CORRECTION FACTORS (ANNUALLY)

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

ICP ID Number: _____ Date: _____

Analyte	Wave-length (nm)	Interelement Correction Factors for: Al	Ca	Fe	Mg	___
Aluminum						
Antimony						
Arsenic						
Barium						
Beryllium						
Cadmium						
Calcium						
Chromium						
Cobalt						
Copper						
Iron						
Lead						
Magnesium						
Manganese						
Mercury						
Nickel						
Potassium						
Selenium						
Silver						
Sodium						
Thallium						
Vanadium						
Zinc						

Comments:

FORM XI (PART 1) - IN

Fig. 14 Form XI-Part 1 (ICP Interelement Correction Factors)

U.S. EPA - CLP

11B
ICP INTERELEMENT CORRECTION FACTORS (ANNUALLY)

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

ICP ID Number: _____ Date: _____

Analyte	Wave-length (nm)	Interelement Correction Factors for: ___	___	___	___	___
Aluminum						
Antimony						
Arsenic						
Barium						
Beryllium						
Cadmium						
Calcium						
Chromium						
Cobalt						
Copper						
Iron						
Lead						
Magnesium						
Manganese						
Mercury						
Nickel						
Potassium						
Selenium						
Silver						
Sodium						
Thallium						
Vanadium						
Zinc						

Comments:

FORM XI (PART 2) - IN

Fig. 15 Form XI Part 2 [ICP Interelement Correction Factors (Annually)]

U.S. EPA - CLP

12
ICP LINEAR RANGES (QUARTERLY)

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

ICP ID Number: _____ Date: _____

Analyte	Integ. Time (Sec.)	Concentration (ug/L)	M
Aluminum			
Antimony			
Arsenic			
Barium			
Beryllium			
Cadmium			
Calcium			
Chromium			
Cobalt			
Copper			
Iron			
Lead			
Magnesium			
Manganese			
Mercury			
Nickel			
Potassium			
Selenium			
Silver			
Sodium			
Thallium			
Vanadium			
Zinc			

Comments:

FORM XII - IN

Fig. 16 Form XII (ICP Linear Ranges, Quarterly)

U.S. EPA - CLP

13
PREPARATION LOG

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Method: ____

EPA Sample No.	Preparation Date	Weight (gram)	Volume (mL)

FORM XIII - IN

Fig. 17 Form XIII (Preparation Log)

U.S. EPA - CLP

14
ANALYSIS RUN LOG

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Instrument ID Number: _____ Method: _____

Start Date: _____ End Date: _____

EPA Sample No.	D/F	Time	% R	Analytes																			
				AL	SB	AS	BZ	CD	CA	CR	CO	CU	FE	PB	MG	MN	HG	NI	SE	AG	AL	TL	V	ZN	CN

Form XIV - IN

Fig. 18 Form XIV (Analysis Run Log)

COVER SHEET

LAB CODE: SWOK

CASE #: _____

SDG #: _____

SAS # (if applicable) _____ ___

CONTRACT #: _____

(check one)

___ ORGANIC SOW 2/00

___ ORGANIC SOW OLM01.0

___ INORGANIC SOW-

___ INORGANIC SOW

___ LOW CONCENTRATION ORGANIC SOW OLC01.0

___ LOW CONCENTRATION INORGANIC SOW ILC01.0

___ HIGH CONCENTRATION ORGANIC SOW 9/00

___ HIGH CONCENTRATION INORGANIC SOW HCIN

THIS DATA PACKAGE CONTAINS:

___ RAS data only

___ SAS data only

___ RAS+SAS data

___ regional request/additional data

___ other (please explain below):

_____

Fig. 19 SMO Package Cover sheet

FULL INORGANICS COMPLETE SDC FILE (CSF) INVENTORY SHEET

LABORATORY NAME SOUTHWEST LAB. OF OKLAHOMA
CITY/STATE BROKEN ARROW, OK

CASE NO. _____ SDC NO. _____
SDC NOS. TO FOLLOW _____ _____ _____
SAS NO. _____

CONTRACT NO. _____
SOW NO. IIM04.0

All documents delivered in the Complete SDC File must be original documents where possible. (Reference Exhibit B, Section II F and Section III D.)

		Page #s From	Page #s To	(Please Check:) Lab	Region
1.	Inventory Sheet (DC-2) (Do not number)			___	___
2.	Cover Page	___	___	___	___
3.	Inorganic Analysis Data Sheet (Form I-IN)	___	___	___	___
4.	Initial & Continuing Calibration Verification (Form IIA-IN)	___	___	___	___
5.	CRDL Standards For AA and ICP (Form IIB-IN)	___	___	___	___
	Blanks (Form III-IN)	___	___	___	___
	ICP Interference Check Sample (Form IV-IN)	___	___	___	___
8.	Spike Sample Recovery (Form Va-IN)	___	___	___	___
9.	Post Digest Spike Sample Recovery (Form Vb-IN)	___	___	___	___
10.	Duplicates (Form VI-IN)	___	___	___	___
11.	Laboratory Control Sample (Form VII-IN)	___	___	___	___
12.	Standard Addition Results (Form VIII-IN)	___	___	___	___
13.	ICP Serial Dilutions (Form IX-IN)	___	___	___	___
14.	Instrument Detection Limits (Form X-IN)	___	___	___	___
15.	ICP Interelement Correction Factors (Form XIA-IN)	___	___	___	___
16.	ICP Interelement Correction Factors (Form XIB-IN)	___	___	___	___
17.	ICP Linear Ranges (Form XII-IN)	___	___	___	___
18.	Preparation Log (Form XIII-IN)	___	___	___	___
19.	Analysis Run Log (Form XIV-IN)	___	___	___	___
20.	ICP Raw Data	___	___	___	___
	Furnace AA Raw Data	___	___	___	___
	Mercury Raw Data	___	___	___	___
22.	Cyanide Raw Data	___	___	___	___
24.	Preparation Logs Raw Data	___	___	___	___
25.	Percent Solids Determination Log	___	___	___	___

FORM DC-2-1

Fig. 20 DC-2-1 Form

		Page #s From	Page #s To	(Please Check:) Lab	Region
24.	Traffic Report	___	___	___	___
	EPA Shipping/Receiving Documents	___	___	___	___
	Airbill (No. of Shipments ___)	___	___	___	___
	Chain-of-Custody Records	___	___	___	___
	Sample Tags	___	___	___	___
	Sample Log-in Sheet (Lab & DCI)	___	___	___	___
	SDC Cover Sheet	___	___	___	___
28.	Misc. Shipping/Receiving Records (list all individual records)				
	Telephone Logs	___	___	___	___
	_____	___	___	___	___
29.	Internal Lab Sample Transfer Records & Tracking Sheets (describe or list)	___	___	___	___
	Sample Tag Summary	___	___	___	___
	_____	___	___	___	___
30.	Internal Original Sample Prep & Analysis Records (describe or list)				
	Prep Records _____	___	___	___	___
	Analysis Records _____	___	___	___	___
	Description _____	___	___	___	___
31.	Other Records (describe or list)				
	Telephone Communications Log	___	___	___	___
	Case Narrative	___	___	___	___
	_____	___	___	___	___

32. Comments:

_____

Completed by (CLP Lab):

_____ (Signature) | Inorganic Data Clerk (Print Name & Title) | _____ (Date)

Audited by (EPA):

_____ (Signature) | _____ (Print Name & Title) | _____ (Date)

FORM DC-2-2

Fig. 21 DC-2-2 Form

SOUTHWEST LABORATORY OF OKLAHOMA, INC. *and*
AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.

TM

Standard Operating Procedure

Inorganic Preventive Maintenance Schedule

Document No.: SWL-IN-103

Rev No. / Date: Rev. 2.0 — 08/18/99

APPROVALS	DATES
Procedure Prepared By	Date
Program Manager or Section Supervisor	Date
Laboratory Safety Officer	Date
Laboratory QA/QC Officer	Date

(Effective Date is 5 calendar days after the last signature above - QA/QC Officer)

Document Status

[ ] Controlled DC No. _____ Issued to:_____ Date:_____ QA:_____

[ ] Official Copy OC No.:_____ Issued to:_____ Date:_____ QA:_____

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INORGANIC PREVENTIVE MAINTENANCE SCHEDULE

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4.0 PRECAUTIONS 1
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9.0 DEFINITIONS 2

Attachments

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Figure 7: Preventive Maintenance Schedule for *Leco CR-12 TOC Analyzer* 9

1.0 PURPOSE AND APPLICATION

1.1 The Inorganic Preventive Maintenance Schedule Logbook, which is the planned periodic and systematic care and servicing of the laboratory equipment. This element prevents, detects, and corrects possible problems leading to equipment failure or erroneous measurements.

1.2 Provide information on all observations, maintenance, problems, or changes in the equipment operation that may affect the quality of data that is produced.

2.0 RESPONSIBILITIES.

The analysts of the various instruments listed in Section 3.0, are responsible to ensure the preventive maintenance is performed on each instrument. The analyst is also responsible to provide information that may affect the quality of data that is produced.

3.0 EQUIPMENT

3.1 The following instruments use the listed Preventive Maintenance Schedule in Figure 1-7.

3.1.1 Thermo Jarrell Ash ICP's — Figure 1.

3.1.2 Leeman Automated Mercury Analyzers — Figure 2.

3.1.3 Lachet Quick-Chem Auto Ion Analyzer — Figure 3.

3.1.4 Graphite Furnace Atomic Absorption Spectrophotometers — Figure 4.

3.1.5 Dionex IC Units — Figure 5.

3.1.6 Shimadzu TOC 5050 — Figure 6.

3.1.7 Leco CR-12 TOC Analyzer — Figure 7.

4.0 PRECAUTIONS.

Refer to current version of the Laboratory Safety Plan, SWL-GA-101.

5.0 PROCEDURE

5.1 Instructions for the Preventive maintenance Schedule

5.1.1 In the "ANALYST" field, enter your initials.

5.1.2 In the "DATE" field, enter the current date.

5.1.3 Perform and check off all "DAILY" maintenance items each day the instrument is used.

5.1.4 Perform and check off all "WEEKLY" maintenance items on one of the following days: Tuesday, Wednesday, or Thursday.

5.1.5 Perform and check off all "MONTHLY" maintenance items in the first 10 days of each month.

5.1.6 Perform and check off all "SEMI(BI)-ANNUALLY" maintenance items in the last 10 days of April and November.

5.2 The following information should be written under the heading of "SERVICE \ MAINTENANCE \ PROBLEMS":

5.2.1 Corrective maintenance, which is any immediate maintenance that is performed because of sudden changes in equipment performances.

5.2.2 Any major replacements, repairs, adjustments, or alignments that have been completed by a service engineer or the analyst.

5.2.3 Minor alignments, adjustments, and item replacements that bring the equipment to a ready state.

5.2.4 Subtle changes or trends in equipment operation that may lead to possible equipment failure or erroneous measurements.

5.2.5 Equipment failures and malfunctions.

5.2.6 Significant events, such as electrical shorts, drops, etc., which could adversely affect performance.

6.0 **RECORDS.** Refer to Figures 1-7 of the various Preventive Maintenance Logs.

7.0 **REFERENCES.** Southwest Laboratory of Oklahoma, Inc. Standard Operating Procedures as listed in text.

8.0 **ATTACHMENTS.** The following figures are Preventive Maintenance Schedules for each of the instruments listed in Section 3.0 Equipment.

8.1 Figure 1: Thermo Jarrell Ash ICP's

8.2 Figure 2: Leeman Automated Mercury Analyzers

8.3 Figure 3: Lachet Quick-Chem Auto Ion Analyzer

8.4 Figure 4: Graphite Furnace Atomic Absorption Spectrophotometers

8.5 Figure 5: Dionex IC Units

8.6 Figure 6: Shimadzu TOC 5050

8.7 Figure 7: Leco CR-12 TOC Analyzer

9.0 **DEFINITIONS.** None Required.

PREVENTIVE MAINTENANCE SCHEDULE

INORGANICS DEPARTMENT

PREV. MAINT. SCHED. (IN103S1.DOC) SWL-IN-103, REV. _____ BOOK # IN103-1-1 PAGE _____

ANALYST: _____ DATE: _____

For **THERMO JARRELL ASH ICP's**

(Circle One: **TJA ET1, TJA ET2, TJA 61E**)

DAILY:

1. Change or replace peristalic pump tubing ☐
2. Check nebulizer and sample lines for blockage or wear ☐
3. Check standard and blank intensities on calibration ☐
4. Check vacuum pressure (61E Trasce only) ☐
5. Check exhaust system ☐
6. Check torch assembly ☐

WEEKLY:

1. Clean torch assembly ☐
2. Using light machine oil — oil autosampler and peristalic pump ☐

MONTHLY:

1. Delete old data file ☐
2. Change vacuum pump oil and clean air filters ☐
3. Clean R.F. Generator filters ☐
4. Remove and clean spray chamber ☐

SEMI-ANNUALLY:

1. Preventive maintenance check from TJA service ☐

SERVICE / MAINTENANCE / PROBLEMS:

_____

_____

_____

_____

_____

_____

_____

VERIFIED: _____

SOUTHWEST LABORATORY OF OKLAHOMA, INC.
1700 West Albany • Broken Arrow, OK 74012 • Office (918) 251-2858 • Fax (918) 251-2599

Figure 1: Preventive Maintenance Schedule for *Thermo Jarrell Ash ICP's*

PREVENTIVE MAINTENANCE SCHEDULE

INORGANICS DEPARTMENT

PREV. MAINT. SCHED. (IN103S2.DOC) SWL-IN-103, REV. _____ BOOK # IN103-2-1 PAGE _____

ANALYST: _____ DATE: _____

For **LEEMAN AUTOMATED MERCURY ANALYZERS**

(Circle One: **PS200A, PS200B**)

DAILY:

1. Inspect pump tubing and change if needed ..... ☐
2. Change magnesium perchlorate drying tube ..... ☐
3. Rinse acid rinse tray with 1:1 HNO_3 and soak overnight in 1:1 HNO_3 ..... ☐
4. Rinse standard test tubes with 1:1 HNO_3 and soak in 1:1 HNO_3 overnight ..... ☐
5. Copy data file to diskette ..... ☐

WEEKLY:

1. Back-up data files to diskette and delete from hard drive ..... ☐

MONTHLY:

1. Clean optical cell and lenses ..... ☐

SERVICE / MAINTENANCE / PROBLEMS:

_____

_____

_____

_____

_____

_____

_____

VERIFIED:_____

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Figure 2: Preventive Maintenance Schedule for *Leeman Automated Mercury Analyzers*

PREVENTIVE MAINTENANCE SCHEDULE

INORGANICS DEPARTMENT

PREV. MAINT. SCHED. (IN103S3.DOC) SWL-IN-103, REV. _____ BOOK # IN103-3-1 PAGE _____

ANALYST: _____ DATE: _____

For LACHAT QUICK-CHEM AUTO ION ANALYZER

DAILY:

1. Clean RAS sampler and sample trays ..... ☐
2. Flush the manifold for approximately 5 minutes while exercising the multi-port valve, first with DI water, then 2N HCL, then again with DI water, before and after run. ..... ☐
3. Back-up all data files to floppy disk and network users files ..... ☐

WEEKLY:

1. Wipe the proportioning pump rollers clean with Isopropanol ..... ☐
2. Clean the proportioning pump rollers by spraying lint-free cloth with silicone spray and, with the pump running, hold against the rollers. ..... ☐

MONTHLY:

1. Replace all flared tubing, then check the o-rings and replace if necessary ..... ☐
2. Delete backed-up data and template files for the previous month from each method ..... ☐
3. Delete obsolete method files from each method and run disk optimizer ..... ☐
4. Zip file for the previous month's data files stored in the network user's file for each method. Then delete the data files ..... ☐
5. Zip file, then delete the previous month's data files stored on floppy disk ..... ☐

SERVICE / MAINTENANCE / PROBLEMS:

_____

_____

_____

_____

_____

_____

_____

VERIFIED: _____

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1700 West Albany • Broken Arrow, OK 74012 • Office (918) 251-2858 • Fax (918) 251-2599

Figure 3: Preventive Maintenance Schedule for *Lachat Quick-Chem Auto Ion Analyzer*

PREVENTIVE MAINTENANCE SCHEDULE

INORGANICS DEPARTMENT

PREV. MAINT. SCHED. (IN103S4.DOC) SWL-IN-103, REV. _____ BOOK # IN103-4-1 PAGE _____

ANALYST: _____ DATE: _____

For GRAPHITE FURNACE ATOMIC ABSORPTION SPECTROPHOTOMETERS

(Circle One: **VAR400A, VAR400B, PE5100B**)

DAILY:

1. Clean all graphite parts with a Kimwipe ☐
2. Clean all Quartz/Sapphire windows ☐
3. Check the tube to see if it needs to be replaced ☐
4. Check to make sure the sampler or injector tip is properly depositing the sample into the tube/platform and check cup/tube alignment ☐
5. Check water levels in the rinse and waste bottles ☐
6. Check wavelength, current, background, bandwidth, lamp alignment, and energy levels of lamps used ☐
7. Compare the absorbance of standards with previous ones and check the characteristic mass to see if they are desirable ☐
8. Clean sample tray and cover ☐
9. Check any lines for air bubbles or obstruction ☐

WEEKLY:

1. Clean all instrument and work place surfaces ☐
2. Clean contact rings with methanol ☐
3. Check water levels in water coolers ☐
4. Check inventory for replacement parts ☐
5. Thoroughly clean injector tip ☐
6. Make sure pipettors are calibrated ☐

BI-ANNUALLY:

1. Preventive maintenance check ☐

SERVICE / MAINTENANCE / PROBLEMS:

_____

_____

_____

_____

_____

_____

_____

VERIFIED: _____

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1700 West Albany • Broken Arrow, OK 74012 • Office (918) 251-2858 • Fax (918) 251-2599

Figure 4: Preventive Maintenance Schedule for *Graphite Furnace Atomic Absorption Spectrophotometers*

PREVENTIVE MAINTENANCE SCHEDULE

INORGANICS DEPARTMENT

PREV. MAINT. SCHED. (IN103S5.DOC) SWL-IN-103, REV. _____ BOOK # IN103-5-1 PAGE _____

ANALYST: _____ DATE: _____

For **DIONEX IC UNITS**

(Circle One: **DX100**, **DX2010i**)

DAILY:

1. Check Nitrogen tank for proper pressure ..... ☐
2. Check Helium tank for proper pressure ..... ☐
3. Inspect eluent tanks for level and pressure ..... ☐
4. Wipe and clean Autosampler ..... ☐
5. Check refrigerator temperature ..... ☐ _____ °C
6. Check printer paper supply ..... ☐
7. Wipe I.C. bench area ..... ☐
8. Wipe instrument, including top ..... ☐
9. Check for liquid leaks ..... ☐

MONTHLY:

1. Delete data from hard drive ..... ☐

SERVICE / MAINTENANCE / PROBLEMS:

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VERIFIED: _____

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Figure 5: Preventive Maintenance Schedule ***Dionex IC Units***

PREVENTIVE MAINTENANCE SCHEDULE

INORGANICS DEPARTMENT

PREV. MAINT. SCHED. (IN103S6.DOC) SWL-IN-103, REV. _____ BOOK # IN103-6-1 PAGE _____

ANALYST: _____ DATE: _____

For SHIMADZU TOC 5050

DAILY:

1. Check ultra high pure air pressure ☐
2. Inspect I.C. reaction vessel/I.C. reagent reservoir ☐
3. Inspect needle wash reservoir ☐
4. Inspect dehumidifier drain vessel water level ☐
5. Inspect humidifier water level ☐
6. Inspect microliter syringe ☐

WEEKLY:

1. Replace 2N HCl ☐
2. Wipe down instrument ☐
3. Wipe down bench area ☐

MONTHLY:

1. Replace T.C. catalyst if sample load was high (regenerate twice before analysis) ☐
2. Inspect T.C. sample injection o-ring (replace quarterly) ☐
3. Inspect teflon o-ring in injection book (replace bi-annually) ☐
4. Inspect sample injection needle to T.C. ☐
5. Reposition silicon tube in I.C. drain pinch valve ☐
6. Inspect o-rings in I.C. reaction vessel injection block (replace quarterly) ☐
7. Inspect sample injection needle for I.C. ☐
8. Inspect sampling needles ☐
9. Prepare new standards ☐
10. Prepare new calibration curve ☐

SEMI-ANNUALLY:

1. Inspect soda lime scrubber (replace annually) ☐
2. Inspect halogen scrubber (replace annually) ☐
3. Call Shimadzu for preventive maintenance service ☐

SERVICE / MAINTENANCE / PROBLEMS:

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Figure 6: Preventive Maintenance Schedule for *Shimadzu TOC 5050*

PREVENTIVE MAINTENANCE SCHEDULE

INORGANICS DEPARTMENT

PREV. MAINT. SCHED. (IN103S7.DOC) SWL-IN-103, REV. _____ BOOK # IN103-7-1 PAGE _____

ANALYST: _____ DATE: _____

For LECO CR-12 TOC ANALYZER

DAILY:

1. Check oxygen tank for proper pressure ..... ☐
2. Check flow rate ..... ☐
3. Check furnace temperature ..... ☐
4. Clean used filter tubes ..... ☐
5. Clean used sample boats ..... ☐

SERVICE / MAINTENANCE / PROBLEMS:

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Figure 7: Preventive Maintenance Schedule for *Leco CR-12 TOC Analyzer*

PREVENTIVE MAINTENANCE SCHEDULE

INORGANICS DEPARTMENT

PREV. MAINT. SCHED. (IN103S1.DOC) SWL-IN-103, REV. _____ BOOK # IN103-1-1 PAGE _____

ANALYST: _____ DATE: _____

For **THERMO JARRELL ASH ICP's**

(Circle One: **TJA ET1, TJA ET2, TJA 61E**)

DAILY:

1. Change or replace peristalic pump tubing ..... ☐
2. Check nebulizer and sample lines for blockage or wear ..... ☐
3. Check standard and blank intensities on calibration ..... ☐
4. Check vacuum pressure (61E Trasce only) ..... ☐
5. Check exhaust system ..... ☐
6. Check torch assembly ..... ☐

WEEKLY:

1. Clean torch assembly ..... ☐
2. Using light machine oil — oil autosampler and peristalic pump ..... ☐

MONTHLY:

1. Delete old data file ..... ☐
2. Change vacuum pump oil and clean air filters ..... ☐
3. Clean R.F. Generator filters ..... ☐
4. Remove and clean spray chamber ..... ☐

SEMI-ANNUALLY:

1. Preventive maintenance check from TJA service ..... ☐

SERVICE / MAINTENANCE / PROBLEMS:

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Standard Operating Procedure

1st and 2nd Data Review of Wetlab Analysis

Document No.: SWL-IN-107

Rev No. / Date: Rev. 1.4 — 10/06/00

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APPROVALS	DATES
Procedure Prepared By	10-13-00 Date
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Laboratory QA/QC Officer	10-20-00 Date

(Effective Date is 5 calendar days after the last signature above - QA/QC Officer)

Document Status

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1ST AND 2ND DATA REVIEW OF WETLAB ANALYSIS

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0 PURPOSE AND APPLICATION

This SOP is designed to provide guidance in the review of wetlab analysis. The items reviewed in any analysis run should not be limited to the list in this SOP. The reviewers should use a critical approach, when reviewing the analysis, to assure SWL clients the best quality data available.

2.0 RESPONSIBILITIES

2.1 The first review will be done by the analyst performing the analysis. In some cases, if the analyst performing the analysis is not available, a peer analyst will perform the first review.

2.2 The second review will be performed by the Inorganic Data Reviewer or an analyst considered proficient in the analysis being reviewed, or a person trained by the Inorganic Program Manager in the review of analysis.

2.3 A Technical review will be performed by the Inorganic Program Manager / or Inorganic Supervisor for all CLP and CLP like data packages. Project Officers review all other packages.

3.0 EQUIPMENT

Not applicable.

4.0 PRECAUTIONS

Not applicable.

5.0 PROCEDURE

5.1 General Requirements - This is a minimum requirement for all Wetlab analysis. For specific QA/QC requirements refer to the individually referenced SOP's, under which the analysis was performed. Several tests do not require a calibration, Initial QC, Continuing QC, and Ending QC. In these cases "NA" will be placed on the check sheet.

5.1.1 The analytical sequence agrees with designated SOP requirements.

5.1.1.1 The instrument calibration consists of the correct number, correct concentration of standards, and at the proper frequency; the R2 value is > 0.995.

5.1.1.2 Initial QC must be checked for completeness and must be within established limits for each analyte analyzed.

5.1.1.3 Continuing QC must be checked for time and frequency restrictions concerning continuing QC samples. In addition, continuing QC samples must be within established limits for each analyte analyzed.

5.1.1.4 Ending QC must be checked for completeness and must be within established limits for each analyte analyzed.

5.1.2 Digestion/Preparation QC-Each digestion batch should contain no more than 20 samples (excluding PB, LC, Duplicate, and MS/MSD samples) and the assigned, unique batch number must agree with the preparation date and intended test code.

5.1.2.1 Preparation Blank (PBW/PBS) - The Prep Blank ID # assigned during digestion agrees on all submitted data, including digestion logs, run logs, and raw data. The Prep Blank must be within established limits for each element analyzed. If, not re-digest and/or re-analyze samples.

5.1.2.2 Laboratory Control Samples (LCS/LDS/LCW/LDW) - The LCS ID # assigned during digestion agrees on all submitted data, including digestion logs, run logs, and raw data. Recovery must be within established limits for each element analyzed; data for duplicate LCS must be within the established limits for each element analyzed. If, not re-digest and/or re-analyze samples.

5.1.2.3 Matrix Spike Samples (MS/MSD) - Spiking levels used must agree with the designated SOP requirements. Recovery must be within established limits for each element analyzed; data for duplicate MS must be within established limits for each element analyzed. If failure, follow individual SOP requirements.

5.1.2.4 Duplicate Samples- Several tests do not require a MS/MSD; in this case a Duplicate will be required for each batch of samples. Data processed for all duplicate samples must agree within the established limits for each element analyzed. If failure, follow individual SOP requirements.

5.1.3 Sample Analysis

5.1.3.1 Sample ID's, as indicate on raw data, agrees with samples listed on Digestion Log (if required).

5.1.3.2 Samples were analyzed within the required Hold Time.

5.1.3.3 Sample concentrations are within the established linear range for each element used. If not, the sample must be diluted and re-analyzed. To assure a valid result, the concentration of the diluted sample must be within a reasonable factor above the established reporting limits. Compare results between the dilution and original sample to verify that results are reasonable. Result must be compared for interfering analytes to assure no false positive or negative results are reported for affected analytes. All dilutions will be reported as #X, where "#" is the dilution factor that the sample was diluted (i.e. 10 fold dilution will be reported as "10X")

5.1.3.4 The absence of carryover is verified. This is performed by reviewing the replicate readings for the sample directly following any sample with a concentration over the linear range of the method. If the concentration of the sample in question is higher than the method MDL or IDL and the % RPD of the replicate readings is greater than 20%, the sample must be re-analyzed. For sample analysis with only one replicate, the sample following the sample over linear range must be re-analyzed.

5.1.4 Run Logs

5.1.4.1 Sample ID's and Client ID's are consistent throughout the raw data, run log, and digestion log.

5.1.4.2 Each page of the run log is initialed & dated by the analyst.

5.1.4.3 The SOP used for analysis is written on the first page of the run log and is appropriate for the samples analyzed.

5.1.4.4 Samples and/or analytes <u>not</u> used are clearly labeled and documented.

5.1.4.5 The Standards Batch ID # or unique SWL Standard ID # are clearly written down to provide traceability of the individual calibration and QC standards.

5.1.4.6 Parameters and methods used are clearly labeled on the run log and agree with the analytes requested.

5.1.5 Automatic Upload Reports from Inorganic Database:

5.1.5.1 Reporting units used are recorded and agree with the matrix of samples.

5.1.5.2 Reporting limits are those specified in the Red book and reporting limits reflect any dilutions made.

5.1.5.3 Ten percent of all data reported by upload reports must be verified against raw data. A check mark is to be placed beside the results that were verified. Place upload report with the episode worksheet once it has been verified by the first and second reviewer.

5.1.6 Manual entry for LIMS:

5.1.6.1 Reporting units used agree with the matrix of samples.

5.1.6.2 Reporting limits are those specified in the Red book and reporting limits reflect any dilutions made.

5.1.6.3 Every result and calculation on the summary page (see individual SOP's for instructions and form) must be verified. Place the summary page with the Episode worksheet.

5.1.6.4 If a method that is normally uploaded automatically requires a manual entry, each result must be written on the worksheet and verified by the second reviewer.

5.2 Non-Conformance Report: Refer to the "Corrective Action" SOP SWL-GA-105 for form. This report will be used for, but is not limited to the following situations:

5.2.1 If a sample or samples in a digestion batch require re-digestion. Enough information should be written to provide a complete understanding why the digestion batch was non-compliant to SWL standards. The report must be given to the Inorganic Supervisor, so the re-digestion can be scheduled into the workload.

5.2.2 If a QC sample failed and due to turn around times, sample volume, etc the samples were not able to be re-digested.

5.2.3 A failed QC sample deemed as acceptable since the sample results were not affected.

5.2.4 Any abnormality observed during the analysis run that might have an effect on sample results. This could include but is not limited to autosampler errors, possible contamination, or items the analyst feels a need to narrate.

5.3 Re-Analysis Form (see Attachment 1): This form must be filled out if the digestion batch is missing any re-analysis that was not performed on the original run. The second reviewer will give this form back to the analyst once it has been verified. It must include, but not limited to dilutions, post-spikes, analyte re-analysis due to QC failure or uncertainty of results, etc.

5.4 "1st and 2nd Review Form" (see Attachment 2): Must be filled out to provide confirmation that the appropriate review steps were performed by the first and second reviewer.

6.0 RECORDS

6.1 Re-analysis Form (Attachment 1) – A form that indicates any re-analysis needed to complete the analysis of a group of samples.

6.2 1st and 2nd Review Form (Attachment 2): Provides confirmation that the appropriate review steps were performed by the first and second reviewer.

7.0 REFERENCES

7.1 *"Inorganic Traceablity of Standards"*, SOP SWL-IN-104, current version.

7.2 *"Corrective Action"*, SOP SWL-GA-105, current version.

7.3 Inorganic SOP's, SWL-IN-300 series, 400 series, 500 series, and 600 series.

8.0 DEFINITIONS

1st review: .....Review of data usually performed by bench analyst.

2nd review: .....Review of the first review by a trained analyst.

Peer analyst: .....An analyst that is proficient in the same analysis that is in question.

Instrument Calibration: .....A series of standards used at the beginning of the analysis run to calibrate the instrument.

Initial QC: .....A series of standards used directly after the calibration to verify the instrument was properly calibrated.

Continuing QC: .....A series of standards used throughout analysis run to verify the instrument is still in calibration.

Final QC: .....A series of standards used at the end of the analysis run to verify the instrument is still in calibration.

PB: .....Preparation Blank (water or soil)

Preparation Blank (water-PBW, soil-LCS): Method blank, consisting of D.I. H_2O, taken through digestion and analysis to check for contamination in the system.

LC: .....Laboratory Control Sample (water or soil)

Laboratory Control Sample (water-LCW, soil-LCS): A spiked sample (usually water) taken through digestion and analysis to check the system's accuracy.

Laboratory Duplicate Control Sample (water-LDW, soil-LDS): A spiked sample (usually water) taken through digestion and analysis to check the system's accuracy and precision.

Matrix Spike (MS): .....A spiked sample taken through digestion and analysis to check the accuracy of the matrix.

Matrix Spike Duplicate (MSD): A spiked sample taken through digestion and analysis to check the accuracy and precision of the matrix.

Duplicate Sample: .....A sample taken through digestion and analysis to check the precision of the matrix.

Carryover: .....A sample is given a false positive result due to a previous high sample. This is most commonly caused by rinse time in-between samples is not sufficient.

Replicate readings: .....More than one reading per sample analysis.

Method Detection Limit (MDL): A detection limit derived from seven samples taken through the entire method.

Typist: LLR

Instrument Detection Limit (IDL): A detection limit derived from seven samples analyzed on three non-consecutive days.

Red Book: .....SWL book that consists of all the MDL/PQL per method for the laboratory.

Raw Data:.....Instrumental Printouts.

Standards Batch ID #: .....Refers to a group of standards used in analysis, refer to SWL-IN-104.

SWL Standard ID #:.....Refers to a unique ID given to a standard, refer to SWL-IN-104.

Episode worksheet: .....Clients samples are divided into episodes, on each episode worksheet there is a list of analysis required and protocol to be used.

Serial Dilution:.....A 5x dilution of a sample in the digestion batch.

9.0 ATTACHMENTS

9.1 Attachment 1: Re-analysis Form

9.2 Attachment 2: 1st and 2nd Review Form

ATTACHMENT 1
Sample Re-Analysis Form

Sample Re-Analysis Form, SWL-IN-107

Sample Re-Analysis Form (107-ATT1.DOC) SWLO rev.# _____

Analysis Performed by: SWL-IN-_____

DigestionBatch ID(s):_____

1st Reviewer:_____ Date:_____

2nd Reviewer:_____ Date:_____

Sample ID	Analyte	Comments / Reason

Comments:_____

ATTACHMENT 2
1st and 2nd Review Form

1st and 2nd Review Form, SWL-IN-107

1st and 2nd Review Form (107-ATT2.DOC) SWLO rev.# _____

Analysis Performed by: SWL-IN-_____ Instrument:_____

File Name:_____ QC Ref. #:_____

DigestionBatch ID(s):_____

Analysis Date:_____

1st Reviewer:_____ Date:_____

2nd Reviewer:_____ Date:_____

Review Items	1st Review (check for confirmation)	2nd Review (check for confirmation)
Analytical Sequence:		
Instrument Calibration, Initial QC, Continuing QC, and Final QC	_____	_____
Digestion/Preparation QC:		
Preparation Blank, Laboratory Control	_____	_____
Matrix Spike, and Duplicate Samples	_____	_____
Sample Analysis:		
Sample ID's,	_____	_____
Required Hold time met	_____	_____
Sample Concentrations within Linear Range	_____	_____
Sample Dilutions labeled correctly	_____	_____
Absence of carryover is verified	_____	_____
Run Logs:		
Sample ID/Client ID agreement, Runlog intialed and dated	_____	_____
Samples/elements not used noted, Elements used clearly labeled	_____	_____
Tracebility of Standards	_____	_____
Automatic Upload Reports		
Units and Reporting limits are correct	_____	_____
10% Review of data is verified	_____	_____
Manual Entry to LIMS		
Units and Reporting limits are correct	_____	_____
100% Review of data is verified	_____	_____
The Following is attached (Y / N):		
Non-Conformance Report	_____	_____
Sample Re-Analysis Form	_____	_____

SOUTHWEST LABORATORY OF OKLAHOMA, INC. *and*
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Standard Operating Procedure

Cation Exchange Capacity of Soils (Sodium Acetate)

Document No.: SWL-IN-212

Rev No. / Date: Rev. 2.2 — 05/16/00

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APPROVALS	DATES
Procedure Prepared By	5-11-00 Date
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Bryan Hicks Laboratory Safety Officer	5-16-2000 Date
Chuck Hosner Laboratory QA/QC Officer	5-16-00 Date

(Effective Date is 5 calendar days after the last signature above - QA/QC Officer)

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CATION EXCHANGE CAPACITY OF SOILS (SODIUM ACETATE)

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1.0 SCOPE AND APPLICATION

1.1 This method is applicable to most soils, including calcareous and non-calcareous soils.

1.2 Cation exchange capacity (CEC) is a measure of the quantity of readily exchangeable cations neutralizing negative charge in the soil.

1.3 The SOP covers USEPA SW-846 "Test Methods for Evaluating Solid Waste Physical/Chemical Methods, Method 9081A, Rev. 1," November 1990, with no deviations.

2.0 METHOD SUMMARY

2.1 The soil sample is mixed with an excess of sodium acetate solution, resulting in an exchange of the 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.

2.2 The concentration of displaced sodium is then determined by ICP and results expressed as milli-equivalents per 100g of soil.

3.0 HEALTH AND SAFETY

3.1 Refer to the current version of the "*Laboratory Safety Plan*", SOP SWL-GA-111.

3.2 Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Wearing gloves, eye protection, and a laboratory coat during extraction and analysis is advisable and should provide the proper protection.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING & STORAGE

4.1 Samples should be collected in glass bottles. All bottles must be thoroughly cleaned and rinsed with reagent water.

4.2 Samples should be stored at 4°C.

4.3 Samples should be analyzed within 6 months of sample collection.

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

5.1 In the saturation step, the exchange sites may not be completely saturated with the saturating cation because other cations in the saturating solution compete for adsorption sites or because the saturating cation's replacing power is insufficient to replace the more strongly adsorbed cations. This error would result in an underestimate of CEC.

5.2 In the washing step, there are four potential sources of error. The adsorbed cation may be removed by hydrolysis and replaced by the H ion. It may be replaced by cations brought into solution in the washing solvent from the dissolution of $CaCO_3$, gypsum, and silicates. Cation exchanges may be lost during decantation because they tend to disperse as the excess electrolyte is removed during washing. Some of the original saturating

solution may be retained in the sediment and subsequently extracted as an exchangeable cation if the washing is incomplete or if salt is retained. All but the last of these error sources cause underestimation's of CEC.

5.3 Interferences can occur during analysis of the extract for sodium content. See the SOP for ICP analysis (SWL-IN-205, *ICP SW3010A/3050B Digestion and SW6010B Analysis*) which discusses the possible interferences.

6.0 EQUIPMENT/APPARATUS

6.1 Thermal-Jarrell Ash ICAP-61 or ICAP-61E Trace optical emission spectrophotometer.

6.2 Centrifuge – to hold small plastic 50ml containers.

6.3 Wrist Action Shaker

6.4 Filter Papers – Whatman 4

6.5 Small Funnels – Disposable plastic

6.6 Top loading balance – capable of weighing to 1g.

7.0 REAGENTS

7.1 Sodium Acetate (NaOAc), 1.0 N: Dissolve 136g of sodium acetate in water and dilute to 1-liter. The pH of this solution should be 8.2. If needed, add a few drops of acetic acid or sodium hydroxide solution to bring the reaction of the solution to pH 8.2.

7.2 Ammonium acetate (NH_4OAc), 1N: Dilute 114ml of glacial acetic acid with water to a volume of approximately 1-liter. Then add 138ml of concentrated ammonium hydroxide and water to obtain a volume of about 1,980ml. 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 deionized water.

7.3 Isopropyl alcohol 99%

7.4 ICP standard and solutions – See SOP SWL-IN-205, "*ICP SW3010A/3050B Digestion and SW6010B Analysis*".

8.0 PROCEDURE

8.1 Weight 4g of medium or fine textured soil or 6g of course textured soil into a 50ml plastic container.

8.2 Add 33ml of 1.0 N NaOAc solution, snap the lid securely, shake it in a wrist action shaker for 5 minutes, and centrifuge until supernatant liquid is clear. Setting 5≅20 minutes on the centrifuge.

8.3 Decant the liquid into waste bucket and repeat step 8.2 three more times.

8.4 Add 33ml of 99% isopropyl alcohol, snap the lid securely, shake it in a wrist action shaker for 5 minutes, and centrifuge until supernatant liquid is clear.

8.5 Decant the liquid into a waste bucket and repeat the procedure in step 8.4 two more times.

8.6 Add 33ml NH_4Oac solution, snap the lid securely, shake it in a wrist action shaker for 5 minutes, and centrifuge until supernatant liquid is clear. Decant the washing through a Whatman 4 filter and disposable plastic funnel into a 100ml volumetric flask.

8.7 Repeat the procedure in step 8.6 two more times.

8.8 Dilute the combined washings to the 100ml mark with ammonium acetate solution. Transfer solution to a 100ml plastic beaker and label them with SWLO ID number. Determine the sodium concentration by ICP analysis

9.0 CALCULATIONS

Calculate the cation-exchange capacity in meq Na/100g as follows:

$$\text{meq Na/100g} = \frac{(\text{mg/l Na})}{(W)\,(23)\,(10)} \times 100$$

Where:

W = weight of soil used for extraction

10.0 QUALITY ASSURANCE/QUALITY CONTROL

10.1 Employ a minimum of one blank per extraction/digestion batch of ≤20 samples to determine if contamination or any memory effects are occurring.

10.2 Employ a laboratory control sample (LCS) and a laboratory control duplicate (LDW) per digestion batch.

10.3 Employ a duplicate sample with each extraction batch.

10.4 Analyze one standard at the detection limit (PQL) with each batch of samples.

10.5 See SOP SWL-IN-205 for ICP Analysis QA/QC.

11.0 WASTE DISPOSAL/POLLUTION PREVENTION.

All samples and chemicals will be disposed of according to the instructions given in SOP SWL-GA-114, "*Hazardous Waste Management Plan*".

12.0 METHOD PERFORMANCE

12.1 The result for the method blank must be below the reporting limit (500meq Na/100ml).

12.2 The control limit for the LCW-LDW is 20% RPD and 80-120% recovery.

12.3 The control limit for the duplicate is 0-35% RPD.

12.4 See SOP-SWL-IN-205 for control limits for ICP analysis.

13.0 REFERENCES

13.1 U.S. EPA. SW-846 "*Test Methods for Evaluating Solid Waste Physical/Chemical Methods. Method 9081A. Revision 1*", *November 1990.*

13.2 Rhoades, J.D. "*Cation-exchange Capacity*" pp. 149-157, in C.A. Black (ed.), Methods of Soil Analysis, Part 2: Chemical and Microbiological Properties. Am. Soc. Agron., Madison, Wisconsin (1982).

14.0 DEFINITIONS

14.1 Sample Batch: ..... Consists of a maximum of twenty samples per matrix.

14.2 Preparation Blank: ..... Method blank taken through extraction, digestion and analysis to check for contamination in the system.

14.3 Laboratory Control Sample (LCS): A spiked sample taken through digestion and analysis to check the system's accuracy and precision.

14.4 Laboratory Duplicate Control Sample (LDS): A spiked sample taken through digestion and analysis to check the system's accuracy and precision.

14.5 Duplicate Sample: ..... A sample taken through extraction, digestion and analysis to check the precision of the matrix.

SOUTHWEST LABORATORY OF OKLAHOMA, INC. *and*
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Standard Operating Procedure

Analysis of Total Organic Carbon in Water

Document No.: SWL-IN-310
Rev No. / Date: Rev. 4.1 — 10/26/00

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APPROVALS	DATES
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Laboratory QA/QC Officer	10-31-00 Date

(Effective Date is 5 calendar days after the last signature above - QA/QC Officer)

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ANALYSIS OF TOTAL ORGANIC CARBON IN WATER

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1.0 SCOPE AND APPLICATION

1.1 This method includes the measurement of organic carbon in drinking, surface, and saline waters, domestic and industrial wastes.

1.2 The method is most applicable to measurement of organic carbon between 1.0-100 mg/L.

1.3 The SOP covers USEPA Method 415.1, SW-846 Method 9060, and Standard Methods 5310B. All QC requirements of each are covered using the stricter requirement where applicable.

2.0 METHOD SUMMARY

Organic carbon in a sample is converted to carbon dioxide (CO_2) by catalytic combustion. The CO_2 formed is measured by nondispersive infrared gas analyzer (NDIR).

3.0 HEALTH AND SAFETY

3.1 Refer to the current version of the "*Laboratory Safety Plan*" SOP (SWL-GA-111).

3.2 Each chemical and sample should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Wearing gloves, eye protection, and a laboratory coat during extraction and analysis is advisable and should provide the proper protection.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

4.1 Sampling and storage of samples in glass bottles is preferable. Sampling and storage in plastic bottles is permissible if it is established that the containers do not contribute contaminating organics to the samples.

4.2 Samples should be acidified with HCl or H_2SO_4 to a pH <2. Store samples at 4°C until time of analysis.

4.3 Holding times are 28 days from sample collection.

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

5.1 This procedure is applicable only to homogenous samples, which can be injected into the apparatus by reproducible means of a microliter-type syringe. The openings of the syringe limit the maximum size of particle, which may be included in the sample.

5.2 Removal of carbonate and bicarbonate by acidification and purging with nitrogen can result in the loss of volatile organic substances.

6.0 EQUIPMENT/APPARATUS

6.1 Shimadzu Total Organic Carbon Analyzer, model TOC-5050 or equivalent.

6.2 Carrier Gas: Zero Grade Air not to contain more than 1ppm CO_2, CO, and HC (hydrocarbon) respectively.

7.0 REAGENTS

7.1 *Distilled water* used in preparation of standards and for dilution of samples should be ultra pure to reduce the carbon concentration of the blank.

7.2 *Potassium hydrogen phthalate, stock solution, 1000mg carbon/liter*: Dissolve 2.125g of potassium hydrogen phthalate (Primary Standard Grade) in distilled water and dilute to one liter. The obtained solution contains 1,000 mg/l of carbon (1,000 C/1) or equivalent to 1,000ppm C.

7.3 *IC Reagent*: 25ml phosphoric acid, 75ml DI water.

7.4 *Humidifier:* Fill humidifier with distilled water to the upper line, add 2 pellets of NaOH, and mix.

7.5 *Working Calibration Standards:* Using the following table, dilute the standards using organic-free water.

mLs of Stock Solution (6.2) per 200mL Final Volume	Conc. of Standard mg/l
0.2	1.0
4.0	20.0
10.0	50.0
20.0	100.0

7.6 *Blank Solution:* Use the same distilled water used for the preparation of the standard solution.

8.0 PROCEDURE

8.1 Calibration

8.1.1 Summary

8.1.1.1 Lower the pH of the standards to <2 with H_2SO_4.

8.1.1.2 Load the standards into the autosampler and purge with zero grade air for 5 minutes.

8.1.1.3 Calibrate instrument starting with high standard and working down towards low standard.

8.1.1.4 Resulting calibration curve must have a correlation coefficient ≥0.995.

8.1.2 Calibration Frequency:

8.1.2.1 A new calibration must be performed on a monthly basis, or if one of the following action items exist (whichever is more frequent):

- When a new Stock standard solution is made.
- When any of the instrument parameters are changed (i.e. combustion, tube, carrier gas flow rate, injection needle, or anything that might change the sensitivity of the instrument).
- If the Independent Calibration Standard (ICV), Continuing Calibration Standard (CCV), Independent Calibration Blank (ICB), or Continuing Blank (CCB) fails their respective control limits.

8.1.3 Procedure

8.1.3.1 Remove gas-line from probe.

8.1.3.2 Bring up "Main Menu": *Press* "#8" (Maintenance Menu). Arrow down to gas-flow (*press* "Enter"). This will turn the gas on. Open front cover of the instrument and adjust the gas-flow all the way open.

8.1.3.3 Place gas line in standard container and sparge for five minutes. Do all standards, cleaning gas line between each standard.

8.1.3.4 Fill standard vials and place in turntable starting with high standard. The setup will be as follows:

CALIBRATION ONE	
100.0	S1
50.0	S2
20.0	S3
1.0	S4

8.1.3.5 Check baseline by *selecting* "#6" (Monitor) and correct if necessary. Press F2 to return to the Main Menu.

8.1.3.6 Next *select* "#9" (Autosampler) and *press* "Enter".

8.1.3.6.1 *Press* "F3" (Edit)

8.1.3.6.2 *Press* "F1" (All Clear)

8.1.3.6.3 *Press* "F2" (All Clear)

8.1.3.7 Arrow over to "C1" (Calibration One). Here you may enter any number one through eighteen and *press* "Enter". The calibration will be stored in that file number.

8.1.3.7.1 Type will be set as "#1" (TC). *Press* "Enter".

8.1.3.7.2 The first standard concentration will be 100.0: *Press* "Enter". Next enter where the standard will be placed. See section 8.1.3.4 above for placement (i.e., the first standard will be S1).

8.1.3.7.3 Range will be set at x5; *press* "Enter".

8.1.3.7.4 Injection volume will be 30ug/l; *press* "Enter".

8.1.3.7.5 Number of injections will be 3; *press* "Enter".

8.1.3.7.6 Max. Number of injections will be set at 5; *press* "Enter".

8.1.3.7.7 SD and CV will be set at 200 and 2.0%.

8.1.3.7.8 "Shift to Origin" should be set to the "OFF" position.

8.1.3.8 *Press* "F2" (Return) to return to autosampler screen.

8.1.3.9 *Press* "F1" (Next).

8.1.3.10 ASI Conditions:

8.1.3.10.1 Rinse.....1

8.1.3.10.2 No. of Needle Washes.....2

8.1.3.10.3 Flow-line washes.....4

8.1.3.10.4 Calibrate before.....2

8.1.3.10.5 Print.....3

8.1.3.10.6 Screen will skip down to:

Key Lock.....2

8.1.3.11 *Press* "F1" (Next).

8.1.3.12 *Press* "Start".

8.2 Sample Analysis

8.2.1 Summary

8.2.1.1 Load samples onto autosampler.

8.2.1.2 Set purge time at 2.0 minutes using zero grade air as purge gas.

8.2.1.3 Analyze the samples using triple injection.

8.2.2 Procedure

8.2.2.1 *Press* "# 8" (Maintenance); *press* "Enter".

8.2.2.2 Arrow down to mechanical check; *press* "Enter".

8.2.2.3 *Press* "ASI".

8.2.2.4 *Press* "AT-R".

8.2.2.4.1 Remove probe wash and clean with soap and water. Rinse with 1:1 HCl. Fill with DI water and replace; *press* "Return"; *press* "End".

8.2.2.5 Remove sample and gas line from probes.

8.2.2.6 Remove gas and sample probe from arm.

8.2.2.6.1 Place both needles in a very mild HCl solution (1:9) and place in sonicator for fifteen minutes.

8.2.2.6.2 Remove from sonicator and rinse.

8.2.2.6.3 Fill container with DI water and sonicate for an additional fifteen minutes. Replace in arm.

8.2.3 Regeneration of IC

8.2.3.1 *Press* "Enter" once regeneration is complete (refill if necessary).

8.2.3.1.1 Arrow down to mechanical check; *press* "Enter".

8.2.3.1.2 Under IC-D *press* "On" and let any excess reagent drain.

8.2.3.1.3 While on move, drain tube up or down to prevent tube being crimped or broken. *Press* "Off"; then *press* "End".

8.2.3.2 Arrow up to regeneration of TC.

8.2.3.2.1 Place 2N HCl in S1 and *press* "Start".

8.2.3.2.2 Remove top lid off of TOC 5050.

8.2.3.2.3 Watch injection to make sure injection is centered and does not splash on sides of combustion tube. If injection is not correct, replace injection needle (see Shimadzu TOC 5050 Operating Manual, page 123).

8.2.3.3 Connect gas and sample lines to proper probe.

8.2.3.3.1 Arrow down to sparge gas flow and *press* "Enter". This turns the gas on.

8.2.3.3.2 Open front cover; set sparge gas to 40ml/min using flowmeter.

8.2.3.3.3 Set carrier gas at 150ml/min.

8.2.3.3.4 Arrow up to sparge gas-flow; *press* "Enter" (this turns off the gas).

8.2.3.3.5 *Press* "F2" to return to Main Menu.

8.2.4 Baseline check

8.2.4.1 Press #6; *press* "Enter".

8.2.4.2 If baseline s zero, *press* "F2". If not, adjust baseline using the optical zero on top of the instrument.

8.2.4.3 *Press* "F2" to return to Main Menu.

8.2.5 Analysis

8.2.5.1 *Press* "#9" (Autosampler); *press* "Enter".

8.2.5.2 *Press* "F3" (Edit).

8.2.5.3 *Press* "F1" (All Clear).

8.2.5.4 *Press* "F2" (All Clear).

8.2.5.5 Choose type of measurement: 1=TC, 2=IC, 3=TOC, 4-NPOC. *Press* "Enter".

8.2.5.6 "1S" is the initial sample. Most often it will be number one, but it can be two, three, and so on.

8.2.5.7 "FS" is final sample and is always a CCB (each run ends with a CCV and a CCB).

8.2.5.8 Arrow over to "F1" and choose "F1" or "F2". "F1" is calibration one; "F2" is the second calibration used for samples that are well above calibration one. *Enter* "#2" (instrument will automatically move to "W" which is number of washes. *Enter* "#4". "NO" is the number of injections: in most cases "#3" will be used. "Max" is the number of injections; in most cases "#5" will be used. If for instance, quads are required it is best to run four out of six. Arrow over to "SP" where sparge gas is set for 2.0 minutes per sample. DI will remain as is.

8.2.5.9 ASI Conditions:

8.2.5.9.1 Rinse..... 1

8.2.5.9.2 Number of needle washes..... 2

8.2.5.9.3 Flow line washes 4

8.2.5.9.4 Calibrate before 2

8.2.5.9.5 Print Info..... 3

8.2.5.9.6 Auto add of acid 1

8.2.5.9.7 Acid volume ..... (auto)

8.2.5.9.8 Rinse after addition of acid .. 1

8.2.5.9.9 Key Lock 2

8.2.5.9.10 Finish or running 3

8.2.5.10 *Press* "Start"

9.0 CALCULATIONS

9.1 Instrument reports all results as mg/L; apply the appropriate dilution factor when necessary.

9.2 Report results and detection limits to 2 significant figures if result is <10.0 mg/L or three significant figures is report is ≥10.0 mg/L.

10.0 QUALITY ASSURANCE/QUALITY CONTROL

10.1 All quality control data should be maintained and available for easy reference or inspection.

10.2 Calibration: See section 8.0.

10.3 Initial Demonstration of Performance:

10.3.1 Linear Calibration Range (LCR) – The LCR must be determined initially and verified every 6 months. The verification on linearity must use a minimum of a blank and three standards. If any verification data exceeds the initial values by ± 10%, linearity must be re-established. If any portion of the range is shown to be nonlinear, sufficient standards must be used to clearly define the nonlinear portion.

10.3.2 Method Detection Limit (MDL) – MDL's must be determined, according to the SWL-GA-113 "*Definition and Determination of Detection Limits*".

10.4 Assessing Laboratory Performance:

10.4.1 A method blank must be distilled with every batch of 20 samples or less. If the value exceeds the reporting limit, laboratory or reagent contamination should be suspected and the samples re-distilled.

10.4.2 Laboratory Control Samples (LCW and LDW) will be distilled with every batch. With a true value of 25.0 mg/l the LCW and LDW have a control limit of 90-110% recovery. If out of control the samples will be re-distilled.

10.4.3 Independent Calibration Verification (ICV) and Continuing Calibration Verification (CCV): this must be a mid-range check standard from an independent source from the calibration standards. It will be run immediately after calibration, a frequency of 10% and at the end of run. Control limits are 90-110% recovery, if failure occurs the system must be re-established and all samples that are not surrounded by CCV's that are in control must be re-analyzed.

10.4.4 Independent Calibration Blank (ICB) and Continuing Calibration Blank (CCB): A blank must be run immediately after each ICV or CCV. The value must be less than the reporting limit; if failure occurs the system must be re-calibrated and all samples that are not surrounded by CCB's that are in control must be re-analyzed.

10.5 Assessing Analyte Recovery and Data Quality

A Matrix Spike (MS) and (MSD) must be performed with every batch at value of 25.0 mg/l level. Data will be flagged if the control of 90-110% is not met.

10.6 Analyze one standard at the detection limit (PQL) with each batch of samples.

11.0 WASTE DISPOSAL/POLLUTION PREVENTION

Refer to the current version of SOP *"Hazardous Waste Management Plan"*, SWL-GA-114.

12.0 METHOD PERFORMANCE

12.1 The current MDL studies are provided in the Red Book. See the Inorganic Program Manager for raw data.

12.2 A method blank must be distilled with every batch of 20 samples or less. If the value exceeds the reporting limit, laboratory or reagent contamination should be suspected and the samples re-distilled.

12.3 Laboratory Control Samples (LCW and LDW) will be distilled with every batch. With a true value of 25.0 mg/l the LCW and LDW have control limit of 90-110% recovery. If out of control the samples will be re-distilled.

12.4 Independent Calibration Verification (ICV) and Continuing Calibration Verification (CCV): this must be a mid-range check standard from an independent source from the calibration standards. It will be run immediately after calibration, a frequency of 10% and at the end of run. Control limits are 90-110% recovery, if failure occurs the system must be re-established and all samples that are not surrounded by CCV's that are in control must be re-analyzed.

12.5 Independent Calibration Blank (ICB) and Continuing Calibration Blank (CCB): A blank must be run immediately after each ICV or CCV.. The value must be less than the reporting limit; if failure occurs the system must be re-calibrated and all samples that are not surrounded by CCB's that are in control must be re-analyzed.

12.6 A Matrix Spike (MS) and (MSD) must be performed with every batch at value of 25.0 mg/l level. Data will be flagged if the control of 90-110% is not met.

13.0 REFERENCES

13.1 U.S. EPA "*Methods for Chemical Analysis of Water and Wastes*", EPA 600/9-91/034, September 1991. Method 415.1

13.2 Instruction Manual, Total Organic Carbon Analyzer Model TOC-5050.

13.3 U.S. EPA SW846 Method 9060, Revision 1, November 1990.

13.4 Standards Methods, 18th Edition, Method 5310B.

14.0 DEFINITIONS

14.1 Material Safety Data Sheet (MSDS): Information provided by commercial vendors for the chemical products prepared by their company. The information on the MSDS includes but is not limited to composition, physical properties, hazard identification, first aid measures, emergency clean up and/or response, exposure and storage.

14.2 Method Detection Limit (MDL): A detection limit derived from seven samples taken through the entire method.

14.3 Practical Detection Limit (PQL): SWL reporting limit based on a factor times the MDL.

14.4 Sample Batch: ..... Consists of a maximum of twenty samples per matrix.

14.5 Preparation Blank (water-PBW): Method blank taken through digestion and analysis to check for contamination in the system.

14.6 Laboratory Control Sample (water-LCW): A spiked sample (usually water) taken through digestion and analysis to check the system's accuracy.

14.7 Laboratory Duplicate Control Sample (water-LDW): A spiked sample (usually water) taken through digestion and analysis to check the system's accuracy and precision.

14.8 Matrix Spike (MS): ..... A spiked sample taken through digestion and analysis to check the accuracy of the matrix. This is designed to provide information about the effect of the sample matrix on the digestion and/or measurement methodology.

14.9 Matrix Spike Duplicate (MSD): A spiked sample taken through digestion and analysis to check the accuracy and precision of the matrix. This is designed to provide information about the effect of the sample matrix on the digestion and/or measurement methodology.

14.10 Duplicate Sample: ..... A sample taken through digestion and analysis to check the precision of the matrix.

14.11 Redbook: ..... A listing of all IDL, MDL, and PQL for the laboratory.

14.12 Bluebook:.....Summary listing of in-house control limits.

14.13 Initial Calibration of Verification Standard (ICV): Prepared from a source of standard solutions other than those used for the calibration solutions and preferably from a different vendor. Used to verify calibration of instrument.

14.14 Continuing Calibration Verification solution (CCV): A mid-range standard used to verify calibration throughout analysis.

14.15 Calibration Blanks (ICB/CCB): A standard blank used to verify calibration throughout analysis.

15.0 ATTACHMENTS

15.1 Figure 1: Front View of the TOC-505 Interior with Front Door Open

ATTACHMENT 1

Figure: Front View of The TOC-5050 Interior With Front Door Open

4. CONSTRUCTION

The front door of the equipment can be opened. LCD, keyboard and sample pan are built in the front door.

The front door need not be opened during measurement. At the time of maintenance and other services, the equipment interior can be accessed easily if front door is opened. Top cover is removed easily. Side panels and bottom panel can be also dismounted by removing screws so that the equipment is very easy to inspect or repair.

Side panels and bottom panel are expected to be opened only by our service staff for repair. If they must be opened by the user, be sure to turn off power switch beforehand.

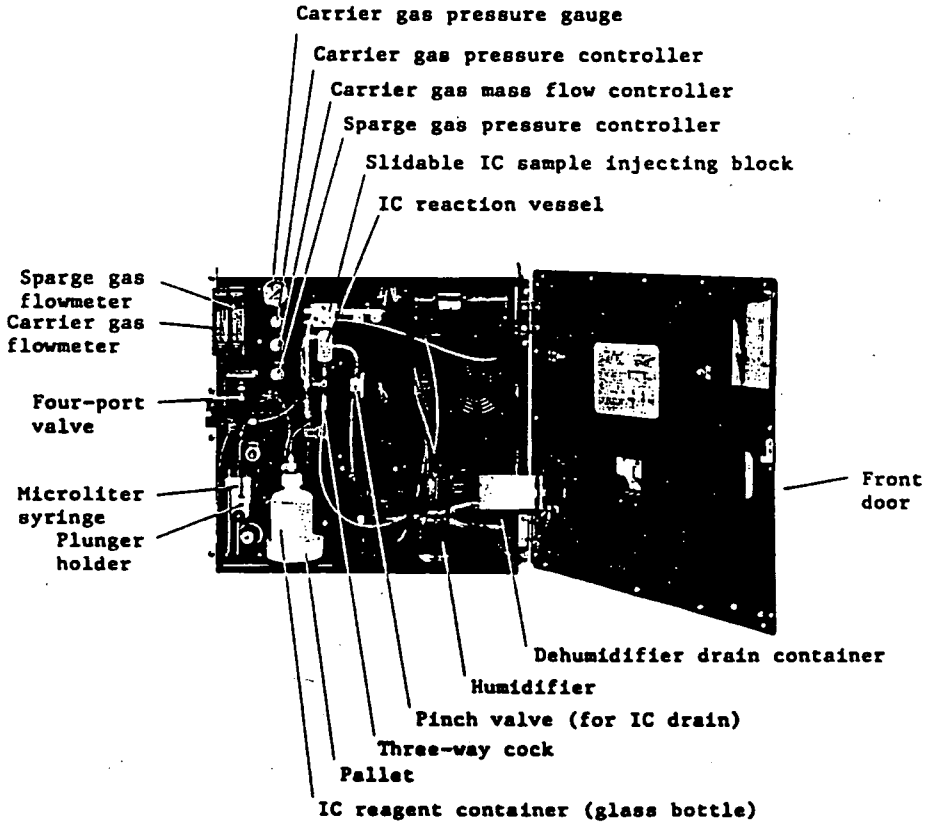

Fig. 4.1 Front View of the TOC-5050 Interior
(Viewed with the Front Door Open)

SOUTHWEST LABORATORY OF OKLAHOMA, INC. *and*
AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.

TM

Standard Operating Procedure

Total Organic Carbon in Soil/Sediment

Document No.: SWL-IN-311

Rev No. / Date: Rev. 4.0 — 05/16/00

OFFICIAL COPY RED STAMP INDICATES ORIGINAL

APPROVALS	DATES
Procedure Prepared By	5-18-00 Date
Program Manager or Section Supervisor	5-18-00 Date
Laboratory Safety Officer	5/19/2000 Date
Laboratory QA/QC Officer	5/18/00 Date

(Effective Date is 5 calendar days after the last signature above - QA/QC Officer)

Document Status

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TOTAL ORGANIC CARBON IN SOIL/SEDIMENT

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1.0 SCOPE AND APPLICATION

1.1 This method describes protocols for the determination of organic carbon in soil or sediment.

1.2 The applicable range of this analysis is 500 to 999.000mg/Kg organic carbon. The Southwest Laboratory of Oklahoma reporting limit is 500mg/Kg.

1.3 Data is reported in mg/Kg on a dry weight basis.

1.4 The SOP covers USEPA SW-846, Method 9060. Rev.1. December. 1996 with no deviations.

2.0 METHOD SUMMARY

2.1 Inorganic carbon from carbonates and bicarbonates is removed by acid treatment.

2.2 The organic compounds are decomposed by pyrolysis in the presence of oxygen.

2.3 The carbon dioxide that is formed is determined by direct non-dispersive infrared detection.

2.4 Water content is determined on a separate portion of sediment.

3.0 HEALTH AND SAFETY

3.1 Refer to current version of "*Laboratory Safety Plan*", SWL-GA-111.

3.2 Each chemical and sample should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. This may be done by wearing gloves, eye protection, and a laboratory coat during analysis.

3.3 Crucibles removed from the furnace are extremely hot. Handle only with tongs while wearing heat-resistant gloves.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING & STORAGE

4.1 Collect sediments in glass jars with Teflon or aluminum foil lined lids.

4.2 Samples must be stored at 4°C.

4.3 Samples must be analyzed within 28 days of collection.

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

5.1 Volatile organics in the soil/sediment samples may be lost in the process of air drying samples as well as decarbonation resulting in a low bias.

5.2 Bacterial decomposition and volatilization of the organic compounds are minimized by maintaining the sample at 4°C, analyzing within the specified holding time, and analyzing the wet sample.

6.0 EQUIPMENT/APPARATUS

6.1 Drying oven maintained at 105°C.

6.2 Analytical Instrument: Leco Model CR-12 Carbon determinator with chlorine-trap and non-dispersive infrared detector.

6.3 Carrier Gas: High Purity Oxygen.

6.4 Integral Alphanumeric Printer.

6.5 Tongs

6.6 Heat resistant gloves.

6.7 Desiccator

6.8 Quartz combustion boats

7.0 REAGENTS

7.1 Distilled water used in preparation of standards and for dilution of samples should be ultra pure to reduce the carbon concentration of the blank.

7.2 Potassium hydrogen phthalate certified A.C.S. grade: 47.05% carbon for instrument calibration.

7.3 Calcium carbonate certified A.C.S. grade: 12% carbon for instrument calibration check.

7.4 Hydrochloric acid solution, 1:1 by volume.

7.5 Chatanooga Shale

8.0 PROCEDURE

8.1 Sample Preparation

8.1.1 Take 0.50 grams of well-mixed sample and place in a quartz combustion boat.

8.1.2 Air-dry this sample in desiccator for minimum of 24 hours.

8.1.3 Add 1:1 HCl solution drop-wise until the effervescence stops.

8.1.4 Dry the samples in a drying oven at 105°C. The sample is now ready for analysis.

8.2 Calibration

8.2.1 Instrument run temp = 2500°C, and standby mode = 1500°C

8.2.2 Select the channel to be calibrated

8.2.3 Add a combustion boat to the built-in analytical balance.

8.2.4 Press tare. Weigh approximately 0.2 grams of potassium hydrogen phthalate in combustion boat.

8.2.5 After the balance has stabilized press enter.

8.2.6 Press "Analyze" and wait for the digital readout to read "Load Furnace". Slide in calibration standard and wait for the result to printout.

8.2.7 Repeat steps 2 through 5 a minimum of three more times.

8.2.8 Press the "SYSTEM UPDATE" key, then the 1 key. The message center will display "CALIBRATE SYSTEM YES/NO".

8.2.9 Press the "YES" key. The message will display "CALIBRATE. BY STD YES/NO".

8.2.10 Press the "YES" key. The printer will print the answer stack for the previous 10 runs.

8.2.11 The message center will display "CARBON STD - % ??? MOD BY KBD", enter the percent carbon contained in the standard sample (47.05%).

8.2.12 Enter the percent carbon obtained in the standard sample (47.05%).

8.2.13 The printer will print the standard entered and the old calibration.

8.2.14 The message will display "SELECT FROM LAST xx RESULT".

8.2.15 Enter the total number of calibration runs to be included in the calibration calculation. After a number is entered, the message center will display one-at-a-time the results for the number of results entered. Answer "YES" or "NO" to each prompt as it appears. The printer will print each answer as the "YES" key is pressed.

8.2.16 After the operator responds to the last prompt, the printer will print the new calibration and the answer stack, recalculated according to the new calibration value.

8.2.17 Analyze the 12% calcium carbonate standard to verify the calibration. The percent recovery must be within ±10% or recalibration will be necessary.

8.2.18 Analyze a blank combustion boat. The result must be below 0.01% or recalibration will be necessary.

8.2.19 The 12% calcium carbonate standard and blank is analyzed after every 10 samples and must meet the above criteria or recalibration and reanalysis of the previous 10 samples will be necessary.

8.3 Sample Analysis

8.3.1 Press the "ID CODE" key. Enter the sample number to be analyzed.

8.3.2 Enter the weight used in the sample preparation and press "ENTER".

8.3.3 Press the "ANALYZE" key. The "PURGE" flow will start immediately. The "LANCE" flow will start later in the analysis cycle.

8.3.4 Wait until the message center display "LOAD FURNACE" and then slide the combustion boat. Immediately after inserting the combustion boat, press the "ANALYZE" key again since the amount of carbon released will be insufficient to start analysis automatically.

8.3.5 When the analysis is complete, the "CARBON" display will indicate the results and the printer will print the value.

8.3.6 Remove the expended combustion boat from the furnace.

8.3.7 Enter results into the "*Soil Total Organic Carbon*" logbook (see Attachment 1).

9.0 CALCULATION

9.1 Multiply % results by 10,000 to obtain TOC in mg/Kg.

9.2 If analysis is done on a wet sample, use the following calculation to convert to dry weight:

$$\text{TOC, mg/Kg} = \frac{\text{TOC wet weight, mg/Kg}}{\text{\% solids}} \times 100$$

10.0 QUALITY ASSURANCE/QUALITY CONTROL

10.1 With each batch of samples a blank (PBS) must be analyzed by treating an empty combustion boat with 1:1 HCl and carrying through the drying procedure with the samples.

10.2 Check the initial calibration (ICV) and every ten samples (CCV) with a calcium carbonate standard as discussed in Section 8.2.

10.3 Analyze a privately prepared sample of Chatanooga Shale, treated the same as the samples, and a duplicate with each batch (LCS/LDS).

10.4 Analyze a blank combustion boat after every ten samples (CCB).

10.5 Analyze one standard at the detection limit (PQL) with each batch of samples.

10.6 A matrix spike (MS) and matrix spike duplicate (MSD) must be analyzed with every batch of samples. Add 0.1 gram of Potassium Hydrogen Phthalate (8.2) after step 8.1.4.

11.0 WASTE DISPOSAL/POLLUTION PREVENTION. Sample waste should be disposed of as per SOP SWL-GA-114, "*Hazardous Waste Management Plan*".

12.0 METHOD PERFORMANCE

12.1 The result of the PBS and CCB must be below the reporting limit (0.05%) or if results are above 10x the blank value the results may be used. If not, the samples must be re-prepared and analyzed.

12.2 The control limits for the ICV and CCV are 90-110% recovery. If outside the control limits re-calibrate and re-analyze the samples.

12.3 The control limits for the LCS/LDS is 85-115% recovery. If recovery is not within control limits, the LCS will be reanalyzed once more. If recovery is still not within control limits the instrument will be re-calibrated and analysis restarted.

12.4 The control limit for MS and MSD is 75-125% recovery, and 0-20% RPD.

13.0 REFERENCES

13.1 Leco Corp, CR-12 Carbon System Instruction Manual, 1987.

13.2 SW-846, Method 9060, Rev. 1, December 1996.

13.3 U.S. EPA, Region II, "*Determination of Total Organic Carbon in Sediment*" by Lloyd Kahn. July 27, 1988.

14.0 DEFINITIONS

14.1 Initial Calibration Verification (ICV): A standard that is from a different source than the calibration standards, and is used to verify the calibration.

14.2 Continuing Calibration Blank (CCB): Empty combustion boat taken through the analysis to check for contamination in the system.

14.3 Continuing Calibration Verification (CCV): Standard used to verify the analytical calibration is still within control.

14.4 Sample Batch: ..... Consists of a maximum of twenty samples per matrix.

14.5 Preparation Blank (PBS):..... Method blank taken through sample preparation steps and analysis to check for contamination in the system.

14.6 Laboratory Control Sample (LCS): A privately prepared sample of known concentration taken through the sample preparation and analysis to check the system's accuracy.

14.7 Matrix Spike (MS): ..... A spiked sample taken through analysis to check the accuracy of the matrix.

14.8 Matrix Spike Duplicate (MSD): A spiked sample taken through analysis to check the accuracy and precision of the matrix.

15.0 ATTACHMENTS

15.1 Attachment 1: Soil Total Organic Carbon Log

ATTACHMENT 1
Soil Total Organic Carbon

SOIL TOTAL ORGANIC CARBON

INORGANICS DEPARTMENT

Soil Total Organic Carbon (IN311F1.DOC) SWL-IN-311, REV. 3.0

Analyst: _____ **Reviewed by:** _____ **Date:** _____

#	Client ID	SWLO NO.	Weight of Sample, gm	TOC, mg/Kg Wet Weight	% Moisture	TOC, mg/Kg Dry Weight
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
21						
22						

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Standard Operating Procedure

pH Electrometric Measurement (Test Codes IN220 and HW200)

Document No.: SWL-IN-405

Rev No. / Date: Rev. 4.3 — 05/16/00

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APPROVALS	DATES
Procedure Prepared By	5-18-00 Date
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Laboratory QA/QC Officer	5/18/00 Date

(Effective Date is 5 calendar days after the last signature above - QA/QC Officer)

Document Status

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pH ELECTROMETRIC MEASUREMENT (TEST CODES IN220 AND HW200)

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1.0 SCOPE AND APPLICATION

1.1 This method is used to measure the pH of aqueous wastes and those multiphase wastes where the aqueous phase constitutes at least 20% of the total volume of the waste. Soils may also be determined after a deionized water extraction.

1.2 Multiple analysis of pH on successive volumes of sample is required. Therefore, this be the case adequate sample size will need to be provided by the client.

1.3 This method will also be used for corrosivity pH.

1.4 The SOP covers USEPA method 150.1 and Standard Methods 4500-H. All QC requirements of each are covered using the stricter requirement where applicable.

1.5 This procedure is based on several methods, (see Section 13); the most stringent quality control of those methods is used in this procedure (see section 10).

2.0 **METHOD SUMMARY.** The pH of the sample is determined electrometrically using a combination electrode. The measuring device is calibrated using a series of standard solutions of known pH.

3.0 **HEALTH AND SAFETY.** Refer to "*Laboratory Safety Plan*", SOP SWL-GA-111.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

4.1 Samples should be analyzed as soon as possible preferably in the field at the time of sampling.

4.2 High-purity waters and waters not at equilibrium with the atmosphere are subject to changes when exposed tot he atmosphere, therefore the sample containers should be filled completely and kept sealed prior to analysis.

4.3 Keep soils refrigerated at 4°C. Analyzed immediately after extraction.

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

5.1 The glass electrode, in general, is not subject to solution interferences from color, turbidity, colloidal matter, oxidants, reductants, or high salinity.

5.2 Sodium error at pH levels >10 can be reduced or eliminated by using a low-sodium-error electrode.

5.3 Coatings of oily material or particulate matter can impair electrode response. These coatings can usually be removed by gentle wiping or detergent washing, followed by rinsing with distilled water. An additional treatment with hydrochloric acid (1:9) may be necessary to remove any remaining film.

5.4 Temperature effects on the electrometric determination of pH arise from two sources. The first is caused by the change in electrode output at various temperatures. This

interference can be controlled with instruments having temperature compensation or by calibrating the electrode-instrument system at the temperature of the samples. The second source of temperature effects is the change of pH due to changes in the sample as the temperature changes. This error is sample-dependent and cannot be controlled. It should, therefore, be noted by reporting both the pH and temperature at the time of analysis.

6.0 EQUIPMENT / APPARATUS

6.1 pH meter: Orion Model 720A Bench Top pH/ISE Meter

6.2 Electrode: Orion Ross. combination, glass body general purpose.

6.3 Magnetic stirrer and Teflon-coated stirring bar.

6.4 Temperature sensor for automatic compensation.

6.5 Electrode: Low sodium - error electrode

7.0 REAGENTS.

pH buffer: Labcraft. accurate within ± 0.01 pH units. Standardized against primary standards of the NIST. Discard after each use.

8.0 PROCEDURE

8.1 Calibration

8.1.1 Connect electrode to meter. Slide the mode switch to pH. The sample LED should light. If not, press Sample.

8.1.2 Choose 4.0 and 10.0 buffers for the calibration of the instrument.

8.1.3 Place electrode into first buffer.

8.1.4 Press Cal. The Cal 1 LED lights indicating that this is the first buffer and a value has not been entered. Wait for ready LED to light, then scroll in the correct value, using á, â keys, then press enter. The display will freeze for 3 seconds. Then Cal 2 LED will light indicating the meter is ready for the second buffer.

8.1.5 Rinse electrode and place into the second buffer. Wait for ready LED to light. Scroll in the correct value and press enter. After the second buffer value has been entered, the sample LED will light.

8.1.6 Analyze a buffer solution of pH 4.0, 7.0, and 10.0. If the results are not within ± 0.05 pH of the true value, recalibrate the meter; otherwise, proceed with analysis of samples. Slope must be 95% or greater; if not, clean the electrode and recalibrate.

8.2 Place the sample in a clean glass beaker using a sufficient volume to cover the sensing elements of the electrodes and to give adequate clearance for the magnetic stirring bar.

8.3 Thoroughly rinse and gently wipe the electrodes prior to measuring pH of samples.

Immerse the electrodes into the sample beaker and gently stir at a constant rate to provide homogeneity and suspension of solids. Note and record sample pH.

8.3.1 Repeat the analysis of pH on successive volumes of sample until values differ by less than 0.1 pH units for each sample and record duplicate analysis on log.

8.4 For soils, weigh 20.0g soil into a 50ml sample vial. Add 20.0ml Type II reagent water, close and shake for 10 minutes. Allow the sample to stand for 1 hour, or until the supernatant is clear (it will sometimes be necessary to filter or centrifuge the sample). Sample can then be analyzed as described above.

8.5 Write-up & Review Procedure

8.5.1 Record all results on the analysis run log (see Attachment 1).

8.5.2 Review Data according to requirements in Section 10.0 of this SOP, and SOP, "*1st and 2nd Data Review of Wetlab Analysis*", SWL-IN-107.

8.5.3 If needed, fill out a "Non-Conformance Report" (see SOP SWL-IN-107).

8.5.4 If needed, fill out a Re-analysis Form (see SOP SWL-IN-107).

8.5.5 A copy of the Analysis Log is then attached to the raw data, and above forms (if needed) are present for a secondary or peer review

8.5.6 Enter results in LIMS and then place a copy of the Analysis Log with each episode worksheet and note on front of worksheet that the test is complete.

9.0 CALCULATIONS. None required.

10.0 QUALITY ASSURANCE / QUALITY CONTROL

10.1 Duplicate samples will be analyzed routinely. Duplicates of each sample are required for analysis.

10.2 Electrodes must be thoroughly rinsed between samples.

10.3 Check standards of buffer 4.0, 7.0, and 10.0 solutions must be analyzed once per 20 samples.

10.4 Analyze one standard at the detection limit (PQL) with each batch of samples.

11.0 WASTE DISPOSAL/POLLUTION PREVENTION. See "*Hazardous Waste Management Plan*", SOP SWL-GA-114.

12.0 METHOD PERFORMANCE

Forty-four analysts in twenty laboratories analyzed six synthetic water samples containing exact increments of hydrogen-hydroxyl ions, with the following results:

pH Units	Standard Deviation pH Units	Accuracy as Bias %	Accuracy as Bias pH Units
3.5	0.10	-0.29	-0.01
3.5	0.11	-0.00	
7.1	0.20	+1.01	+0.07
7.2	0.18	-0.03	-0.002
8.0	0.13	-0.12	-0.01
8.0	0.12	+0.16	+0.01

13.0 REFERENCES

13.1 National Bureau of Standards. Standard Reference Material Catalog 1986- 87, Special Publication 260.

13.2 U.S. EPA SW846, 3rd Edition, December 1997, Method 9045C/9040B

13.3 Methods for Chemical Analysis of Water and Wastes, Method 150.1

13.4 Standard Methods, 4500-H

13.5 Selected SWOK SOP's as listed in text.

14.0 DEFINITIONS. None required.

15.0 ATTACHMENTS

Attachment: pH Analysis Log

ATTACHMENT 1
pH Analysis Log

pH ANALYSIS LOG

INORGANICS DEPARTMENT

pH ANALYSIS LOG (IN405F1.DOC) SWL-IN-405. REV. _____ BOOK # IN405-1-___ PAGE _____

Analyst: _____ Date: _____ Prep Batch ID: _____

Start Time: _____ End Time: _____ Buffer 4.0 SWOK ID: _____

Buffer 7.0 SWOK ID: _____

Buffer 10.0 SWOK ID: _____

#	SWL ID #	Initial Wt/vol (g) / (ml)	Volume Final (ml)	Final Value pH	Diff.	Comments
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						

REVIEWED BY: _____

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Typist: LLR

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Standard Operating Procedure

Analysis of Inorganic Chloride

Document No.: SWL-IN-505

Rev No. / Date: Rev. 2.2 — 05/17/00

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ANALYSIS OF INORGANIC CHLORIDE

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1.0 SCOPE AND APPLICATION

1.1 This method is used to measure the inorganic anion chloride (Cl^-) in drinking, surface and saline waters, domestic and industrial wastes. It covers USEPA method 325.3 and Standard Methods 4500-Cl^- C. All QC requirements of each are covered using the stricter requirement where applicable.

1.2 The method is suitable for all concentrations of Chloride: however, in order to avoid large titration volume, a sample aliquot containing not more than 10 to 20 mg Chloride per 50 ml is used.

2.0 METHOD SUMMARY

— An acidified sample is titrated with mercuric nitrate in the presence of mixed diphenylcarbazone-bromophenol blue indicator. The end point of the titration is the formation of the blue-violet mercury diphenylcarbazone complex.

3.0 HEALTH AND SAFETY

— Refer to the current version of "Laboratory Safety Plan", SOP.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

4.1 Samples shall be collected in pre-cleaned plastic or glass containers.

4.2 No sample preservation is required.

4.3 Samples must be analyzed within 28 days of collection.

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

5.1 Chromate and Ferric ions interfere when present in excess of 10mg/L. Add 2 ml of fresh hydroquinone solution (see section 7.6).

5.2 Sulfite interference can be eliminated by oxidizing the 50ml of sample solution with 0.5ml of H_2O_2 (see section 7.5).

6.0 EQUIPMENT/APPARATUS

6.1 Erlenmeyer Flask, 250ul

6.2 Microburet, 10ul

7.0 REAGENTS

7.1 ASTM Type II Water

7.2 Standard Sodium solution, 0.025N - Dissolve 1.4613grams ± 0.0002 g of Sodium Chloride (dried to 600° C for 1 hr.) in Chloride-free water in a 1-liter volumetric flask and dilute to the mark.

7.3 NitricAcid(HNO_3) Solution - Add 3.0 ml concentrated nitric acid to 997 ml of Type II water.

7.4 Sodium Hydroxide (NaOH) Solution - (10g/l): Dissolve approximately 10g of NaOH in Type II water. and dilute to 1L.

7.5 Hydrogen Peroxide - (30% H_2O_2)

7.6 Hydroquinone Solution(10g/L) - Dissolve 1 g of purified hydroquinone in water in a 100-ml volumetric flask and dilute to the mark.

7.7 Mercuric Nitrate Titrant (0.141N) - Dissolve 24.2g $Hg(NO_3)_2$ H_2O in 900 ml of Type II water acidified with 5.0 ml concentrated HNO_3 in a 1 liter volumetric flask and dilute to the mark with Type II water. Filter. if necessary. Standardize against standard sodium chloride solution (see section 7.2) using the procedures in Section 8.0. Adjust to exactly 0.141N and check. Store in a dark bottle. A 1.00ml aliquot is equivalent to 5.00 mg of chloride.

7.8 Mercuric Nitrate Titrant(0.025 N) - Dissolve 4.2830 g $(HgNO_3)_2$ H_2O in 50 ml of Type II water acidified with 0.50 ml of concentrated HNO_3 in 1 liter volumetric flask and dilute to the mark with Type II water. Filter if necessary. Standardize against standard sodium chloride solution (see section 7.2) using the procedures in Section 8.0. Adjust to exactly 0.025N and check. Store in a dark bottle.

7.9 Mercuric Nitrate Titrant (0.0141N) - Dissolve 2.4200 g $(HgNO_3)_2$ in 25 ml of Type II water acidified with 0.25 ml of concentrated HNO_3 in 1 liter volumetric flask and dilute to the mark with Type II water. Filter, if necessary. Standardize against standard sodium chloride (see section 7.2) using the procedures in section 8.0. Adjust to exactly 0.0141N and check. Store in a dark bottle. A 1ml aliquot is equivalent to 500ug of chloride.

7.10 Mixed Indicator Reagent - Dissolve 0.5 g crystalline diphenylcarbozone and 0.05g bromophenol blue powder in 75 ml 95% ethanol in a 100 ml volumetric flask and dilute to the mark with 95% ethanol. Store in a brown bottle and discard after 6 months.

8.0 PROCEDURE

8.1 Place 50 ml of sample in a vessel for titration. Determine an indicator blank on 50ml chloride-free water using section 8.6.

8.2 Add 5 to 10 drops of mixed indicator reagent (see section 7.10). Shake or swirl solution.

8.3 If a blue-violet or red color appears. add HNO_3 solution (see section 7.3) drop-by-drop until the color changes to yellow.

8.4 If a yellow or orange color forms immediately on addition of the mixed indicator, add NaOH solution (see section 7.4) drop-by-drop until the color changes to blue-violet; then add HNO_3 solution (see section 7.3) drop-by-drop until the color changes to yellow.

8.5 Add 1ml excess HNO_3 solution (see section 7.3).

8.6 Titrate with 0.025 N Mercuric Nitrate Titrant (see section 7.8) until a blue-violet color persists throughout the solution. If the volume of titrant exceeds 10ml or is less than 1ml. use the 0.141N or 0.0141N Mercuric Nitrate solutions. respectively. If necessary, take a smaller sample aliquot.

9.0 CALCULATIONS

9.1 To determine the concentration of chloride in the sample. use the following equation:

$$\text{mg Chloride/liter} = \frac{(A-B)N \times 35.450}{\text{ml of sample}}$$

where: A = ml titrant for sample
B = ml titrant for blank
N = normality of mercuric nitrate titrant

10.0 QUALITY ASSURANCE/QUALITY CONTROL

10.1 All quality control data should be maintained and available for easy reference and inspection.

10.2 Analyze one standard at the detection limit (PQL) with each batch of samples.

10.3 Employ a minimum of one blank per sample batch to determine if contamination has occurred.

10.4 A laboratory control sample (LCS) and a laboratory control duplicate (LDS) must be run for each sample batch. Recovery should be within + 20% of the true value.

10.5 A matrix spike (MS) and matrix spike duplicate (MSD) will be done at a frequency of 1 MS/MSD per batch or 20 or fewer samples.

11.0 **WASTE DISPOSAL / POLLUTION PREVENTION**— All chemicals and samples will be disposed of according to the instructions given in SOP, SWL-GA-114, "Hazardous Waste Management Plan".

12.0 METHOD PERFORMANCE — Recovery of the LCS and LDS should be within 20% of the true value with a 20%RPD.

13.0 REFERENCES

13.1 Use EPA SW846. 3rd Edition. Sept. 1994, Method 9252A

13.2 EPA Method for Chemical Analysis of Water and Wastes. Method 325.3.

14.0 DEFINITIONS

14.1 Method Blank: ..... Distilled water sample carried through the analysis to ensure contamination is not present.

14.2 Laboratory Control Sample (LCS): ..... A spiked sample taken through the analysis to check the methods accuracy.

14.3 Laboratory Duplicate Control Sample (LDS):.. A spiked sample taken through the analysis to check the methods accuracy and precision.

14.4 Sample Batch: ..... Consists of a maximum of twenty samples per matrix.

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Standard Operating Procedure

Analysis of Total, Suspended, Dissolved, Fixed and Volatile Solids

Document No.: SWL-IN-600

Rev No. / Date: Rev. 4.0 — 06/02/00

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Program Manager or Section Supervisor	6-15-00 Date
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Laboratory QA/QC Officer	6/15/00 Date

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ANALYSIS OF TOTAL, SUSPENDED, DISSOLVED, FIXED AND VOLATILE SOLIDS

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1.0 SCOPE AND APPLICATION

1.1 This SOP is applicable for drinking, surface and saline water, domestic and industrial wastes, activated sludge, sewage, or bottom sediments.

1.2 The practical range of the determinations vary from 4mg/l — 20,000mg/l for Total Suspended Solids (TSS) to 10mg/l — 20,000mg/l for Total Solids (TS) and Total Dissolved Solids (TDS), and 11mg/l at 170mg/l for Total Volatile Solids.

1.3 The SOP covers USEPA Methods 160.1-160.4 and Standard Methods 2540B-E. All QC requirements of each are covered using the stricter requirement where applicable.

2.0 METHOD SUMMARY

2.1 Total Solids — A well-mixed sample is dried to constant weight at 103°C to 105°C.

2.2 Total Suspended Solids and Total Dissolved Solids — A well-mixed sample is filtered through a glass fiber filter. The residue retained on the filter is dried to constant weight at 103°C to 105°C for Total Suspended Solids, while the filtrate is dried to constant weight at 180°C for Total Dissolved Solids.

2.3 Total Volatile Solids — The residue obtained from the determination of total, filterable or non-filterable residue is ignited at 550°C in a muffle furnace. The loss of weight on ignition is reported as mg/l volatile residue.

3.0 HEALTH AND SAFETY — All samples should be considered hazardous and handled as set forth in SOP, SWL-GA-111 "*Laboratory Safety Plan*".

4.0 SAMPLE PRESRVATION, CONTAINERS, HANDLING AND STORAGE

4.1 Non-representative particulate such as leaves, sticks, fish and lumps of fecal matter should be excluded from the sample.

4.2 Preservation of the sample is not practical; analysis should begin as soon as possible. Refrigeration to 4°C minimizes microbiological decomposition of the solids.

4.3 Samples may be collected in pre-cleaned glass or plastic containers.

4.4 Holding time is 7 days from sample collection.

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

5.1 Highly mineralized waters containing significant concentrations of calcium magnesium, chloride and/or sulfate may be hygroscopic and will require prolonged drying, desiccation and rapid weighing of total dissolved solids.

5.2 Too many residues in the evaporating dish may form a water-trapping crust, which will entrap water that will not be driven off during drying. Residue should be limited to about 200mg for total dissolved solids.

5.3 Samples containing high concentrations of bicarbonate will require careful and possibly prolonged drying at 180°C for dissolved solids to ensure that all the bicarbonate is converted to carbonate.

5.4 For the analysis of total suspended solids, samples high in dissolved solids such as saline waters, brines and some waters, may be subject to a positive interference. Care must betaken in selecting the filtering apparatus so that washing of the filter and any dissolved solids in the filter minimizes this potential interference.

5.5 The volatile solids test is subject to many errors due to loss of water of crystallization, loss of volatile organic matter prior to combustion, incomplete oxidation of certain complex organics, and decomposition of mineral salts during combustion. The results should not be considered an accurate measure of organic carbon in the sample, but may be useful in the control of plant operations. The principal source of error in the determination is failure to obtain a representative sample.

6.0 EQUIPMENT/APPARATUS

6.1 Evaporating dishes, porcelain: 100ml volume.

6.2 Filter supports: Gooch Crucible and holder.

6.3 Vacuum flask, 1L.

6.4 Gooch Crucibles, 25ml.

6.5 Drying oven, 103°-105°C

6.6 Drying oven, 180°C.

6.7 Analytical balances, Mettler H80 or S/P180.

6.8 Desiccator

6.9 Muffle furnace, 550°C

7.0 REAGENTS — Not Applicable

8.0 PROCEDURE

8.1 Total Solids

8.1.1 Prepare the evaporating dishes by heating the clean dish to 103°C to 105°C for one hour. Cool in desiccator and store until needed.

8.1.2 Weigh the dish on an analytical balance, recording the weight in the Solids Logbook (see Attachment) to four decimal places.

8.1.3 Shake the sample vigorously, and rapidly transfer 100ml to the evaporating dish by means of a 100ml graduated cylinder. Rinse the graduated cylinder with three portions of ASTM Type II water, putting each rinse into the evaporating dish.

8.1.4 Transfer the evaporating dish to the drying oven and evaporate to dryness without splattering. Dry the evaporated sample for at least 1 hour in and oven at 103°C to 105°C. Cool the dish in a desiccator to balance temperature and weigh. Record the weight to four decimal places in the solids logbook.

8.1.5 Repeat the cycle of drying, cooling, desiccating and weighing until a constant weight is obtained, or until weight loss is less than 4% of previous weight or 0.5mg, whichever is less.

8.2 Total Dissolved Solids

8.2.1 Prepare the glass fiber disc by placing the disc on the bottom of a Gooch Crucible. While vacuum is applied, wash the disc with three successive 20ml volumes of distilled water. Remove all traces of water by continuing to apply vacuum. Discard washings.

8.2.2 Prepare the evaporating dish by heating the dish to 180°±2°C for one hour. Cool in a desiccator and store until needed.

8.2.3 Immediately before use weigh the dish on an analytical balance and record the weight to four decimal places in the solids logbook.

8.2.4 Assemble the filtering apparatus and begin suction. Shake the sample vigorously and rapidly transfer 100ml to the funnel by means of a 100ml-graduated cylinder.

8.2.5 Filter the sample through the glass fiber filter. Rinse the cylinder and Gooch crucible with three portions of distilled water and continue to apply vacuum for about 3 minutes after filtration is complete to remove as much water as possible.

8.2.6 Transfer the filtrate to the weighed evaporating dish and evaporate to dryness in the oven at a temperature less than 100°C.

8.2.7 When the dish is dry, increase temperature to 180°C and heat for at least one hour at 180°C ± 2°C cool in a desiccator to balance temperature and weigh. Record weight to four decimal places in the solids logbook.

8.2.8 Repeat cycle of drying, cooling, desiccating and weighing until a constant weight is obtained or until weight loss is less than 4% of previous weight or 0.5mg, whichever is less.

8.2.9 For North Carolina samples repeat the procedure starting at 8.2.4 if <4.0mg/L result is obtained: repeat one more time (3 times maximum) if necessary. If >200mg/L a reduced volume must be analyzed beginning with 8.2.1.

8.3 Total Suspended Solids

8.3.1 Insert a glass fiber filter into the bottom of a Gooch crucible with the wrinkled surface up.

8.3.2 Apply vacuum, and then wash the filter with three successive 20ml volumes of distilled water. Remove all traces of water by continuing to apply vacuum after water has passed through.

8.3.3 Place the crucible and filter in a drying oven and dry at 103-105°C for at least one hour. Remove to a desiccator and store until needed.

8.3.4 Immediately before use weigh the crucible and filter on an analytical balance. Handle the crucible with forceps or tongs only. Record the weight of the crucible and filter to four decimal places in the solids logbook.

8.3.5 Assemble the filtering apparatus and begin suction. Wet the filter with a small volume of distilled water to seat it against the fritted support.

8.3.6 Shake the sample vigorously and transfer 100ml of the sample to the filter using a graduated cylinder. If the sample has a lot of particulate matter the analyst should use their judgement on how small of volume to use. Remove all traces of water by continuing to apply vacuum after sample has passed through.

8.3.7 With the suction on, wash the graduated cylinder and Gooch crucible wall with three portions of distilled water allowing complete drainage between washing. Remove all traces of water by continuing to apply vacuum after water has passed through.

8.3.8 Using tongs, remove the crucible and filter and place in a drying oven at 103-105°C for at least one hour. Cool in a desiccator to balance temperature, then weigh and record weight in solids logbook.

8.3.9 Repeat the cycle of drying, desiccating and weighing until a constant weight is obtained or until the weight loss is less than 4% of the previous weight or 0.5mg, whichever is less.

8.3.10 For North Carolina Samples repeat the procedure starting at 8.3.4 when results are <4.0mg/L: repeat one more time (3 times maximum) if necessary. If >200mg/L a reduced volume must be analyzed beginning with 8.3.1.

8.4 Total Volatile Solids

8.4.1 Ignite residue produced by the filterable and non-filterable solids, to constant weight in a muffle furnace at a temperature of 550°C. Have furnace up to temperature before inserting sample. Usually, 15 to 20 minutes ignition are required for 200mg residue. However, more than one sample and/or heavier residues may overtax the furnace and necessitate longer ignition times.

8.4.2 Let dish or filter disk cool partially in air until most of the heat has been dissipated. Transfer to a desiccator for final cooling in a dry atmosphere. Do no overload desiccator.

8.4.3 Weigh dish, or disk as soon s it has cooled to balance temperature.

8.4.4 Repeat cycle of igniting, cooling, desiccating, and weighing until a constant weight is obtained or until weight change is less than 4% or 0.5mg, whichever is less. Duplicate determinations should agree within 5% of their average.

8.5 For Drying Ink on new evaporating dishes or gooch crucibles:

8.5.1 Put dishes or crucibles in muffle furnace.

8.5.2 Heat furnace to a temperature of 600°C.

8.5.3 Heat dishes or crucibles for 1 hour at 600°C.

8.5.4 Let dishes or crucibles cool, then prepare as indicated at the beginning of each test procedure.

9.0 CALCULATIONS

9.1 Total Solids:

$$\text{Total Solids, mg/l} = \frac{(A - B) \times 1000}{C}$$

Where:
A = Weight of evaporating dish + residue in mg.
B = Weight of evaporating dish in mg.
C = ml of sample used

9.2 Total Dissolved Solids

$$\text{Total Dissolved Solids, mg/l} = \frac{(A - B) \times 1000}{C}$$

Where:
A = Weight of evaporating dish + residue in mg.
B = Weight of evaporating dish in mg.
C = ml of sample used

9.3 Total Suspended Solids

$$\text{Total Suspended Solids, mg/l} = \frac{(A - B) \times 1000}{C}$$

Where:
A = Weight of filter and crucible + residue in mg.
B = Weight of filter and crucible in mg.
C = ml of sample used

9.4 Total Volatile Solids

Where: A = Weight of evaporating dish + residue before ignition in mg

$$\text{Total Volatile Solids (mg/l)} = \frac{(A - B) \times 1000}{C}$$

B = Weight of evaporating dish in mg after ignition
C = ml of sample used

10.0 QUALITY ASSURANCE/QUALITY CONTROL

10.1 A blank of 100mls of distilled water must be run with every batch of ≤20 samples.

10.2 A Laboratory Control Sample (LCS) must be analyzed with every batch of ≤20 samples.

10.3 A duplicate sample will be analyzed with every batch of ≤20 samples.

10.4 Filter the QC samples after filtering each batch of samples to show that there is no contamination or carry-over between samples.

10.5 Analyze one standard at the detection limit (PQL) with each batch of samples.

11.0 WASTE DISPOSAL/POLLUTION PREVENTION

— All samples will be disposed of as instructed in the SOP SWL-GA-114, "*Hazardous Waste Management Plan*".

12.0 METHOD PERFORMANCE

12.1 Blanks must be less than the reporting limit of 4mg/l for total suspended solids and 10mg/l for total and dissolved solids, and 11mg/l for total volatile solids.

12.2 The LCS must be ±15% recovery of the true value.

13.0 REFERENCES

13.1 USEPA, Methods for Chemical Analysis of Water and Wastes, Methods 160.1, 106.2, 160.3, and 160.4.

13.2 Standard Methods for the Examination of Water and Wastewater, 19th Edition, Methods 2540B, 2540C, 2540D, and 2540E.

14.0 DEFINITIONS

14.1 Solids: .....Matter suspended or dissolved in water or wastewater. Total Solids includes "Total Suspended Solids", the portion of total solids retained by a filter, "Total Dissolved Solids", the portion that passes through the filter, and "Total Volatile Solids", the

residue produced by total solids or total dissolved solids ignited to a constant weight in a muffle furnace.

14.2 Sample Batch: .....Consists of a maximum of twenty samples per matrix.

14.3 Blank: .....Method Blank, consisting of 100ml of distilled water taken through the analysis to check for contamination.

14.4 Laboratory Control Sample: A sample of known concentration taken through the analysis to check the method accuracy.

14.5 Sample Duplicate: .....A sample taken through the analysis to check the matrix effect on method precision.

15.0 ATTACHMENTS

Attachment 1: Solids Logbook

ATTACHMENT 1
Solids Logbook

Solids Logbook, SWL-IN-600

Solids Logbook (IN600A1.doc) SOP SWL-IN-600, Rev. 3.2 Verified: ____ Book # IN-600-SldLog- ____

Type of Analysis (circle): TS, TDS, TSS, TVS, TVSS **Date/Time In:** _____ **Date/Time Out:** _____ **Batch ID:** _____

Analyst Signature: _____ **Temp. In:** _____ **Temp. Out:** _____ **Page** _____ **of Book** _____

Calculations: TS, TDS, TSS = $\frac{(A-B) * 1000}{C}$ TVS, TVSS = $\frac{(A-D) * 1000}{C}$ Fixed Solids = $\frac{(D-B) * 1000}{C}$

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Standard Operating Procedure

Sonicator Tuning

Document No.: SWL-OE-100

Rev No. / Date: Rev. 2.1 — 10/01/99

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Harry M Borg Procedure Prepared By	10-4-99 Date
Harry M Borg Program Manager of Section Supervisor	10-4-99 Date
Chuck Hoover Laboratory Safety Officer	10-4-99 Date
Chuck Hoover Laboratory QA/QC Officer	10-4-99 Date

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SONICATOR TUNING

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1.0 SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to provide Guidance in the tuning of the High Frequency Sonicator used for extraction of semivolatile organic in solid samples.

2.0 METHOD SUMMARY. N/A

3.0 HEALTH AND SAFETY

Loose tuning probes will generate a loud piercing sound.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING & STORAGE

Sonication parts should be stored in a clean environment to decrease the potential for laboratory contamination.

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

5.1 If the instrument has been left in a very cold environment for a prolonged period of time, do not operate until it has reached room temperature.

5.2 The electrical line cord is equipped with a 3-prong grounding plug. Do not under any circumstances remove the grounding prong. The plug must be plugged into a mating 3-prong grounding type outlet.

5.3 Never assemble or disassemble a probe by holding the converter in a vise.

5.4 When using a microtip, never allow the tip to vibrate in air for more than 10 seconds and do not set the OUTPUT CONTROL (6) above "5". Ignoring these instructions will cause the microtip to fracture.

5.5 Do not operate the power supply unless it is connected to the converter(s).

5.6 High voltage is present in the power supply. Do not operate with the cover off.

5.7 Never allow liquid to spill into the converter. Do not use cup horn without a splash shield.

5.8 Do not allow the microtip to vibrate in air for more than 10 seconds.

5.9 When using a microtip or extender, never exceed MICROTIP LIMIT "5" on OUTPUT CONTROL (6).

5.10 Do not allow the vibrating microtip to come in contact with anything but the solution.

5.11 Do not operate the converter in an explosive or caustic atmosphere.

6.0 EQUIPMENT/APPARATUS

6.1 Sonicator (Vibra cell Model VC602) or equivalent, with ¾ inch probe or 4-Post micro-probe.

6.2 Sonicator Tuning Log.

7.0 REAGENTS. N/A

8.0 PROCEDURE

Sonicator Tuning — Each time a new converter, probe, cup horn, tip, microtip, or accessory is used, the power supply is tuned using the following procedure:

8.1 Move the switch above the tuning control to the "DOWN POSITION".

8.2 Ensure that the probe or microtip is not immersed in the solution and that it does not come in contact with anything. If a cup horn or flow through cell is used, make sure that it does not contain any water.

8.3 Set **timer** and **hold.**

8.4 Set "OUTPUT CONTROL" to "10" (to "4" when using a microtip or extender).

CAUTION: When using a microtip, never allow the tip to vibrate in air for more than 10 seconds, and do not set the OUTPUT CONTROL above "5". Ignoring these instructions will cause the microtip to fracture.

8.5 Momentarily hold down "ON/ OFF/ TUNE" switch to "TUNE" and rotate the tuning control clockwise or counterclockwise until a minimum (not maximum) reading (usually less than 20) is obtained on the power monitor. If minimum reading (sometimes referred to as null) cannot be obtained, it is possible that the probe, sup horn, tip, or microtip is loose or out of resonance, or that the power supply or converter requires servicing. A loose probe will usually generate a loud piercing sound.

NOTE: If minimum reading cannot be obtained, check unit without the probe to ascertain whether the power supply or probe is at fault.

8.6 Set "OUTPUT CONTROL" to "4".

8.7 Release "ON/OFF/TUNE" switch.

8.8 With a dual 500 watt Ultrasonic Processor, if two converters are going to be used simultaneously, connect the second converter cable to connector.

8.9 Document all tuning on the Sonicator Tuning Log (see Attachment 1), located in the *Maintenance Zone LogBook*, SWL-GA-128.

9.0 CALCULATIONS. N/A

10.0 QUALITY ASSURANCE/QUALITY CONTROL

See Control Limits (Sonicator Log in Maintenance Zone Book).

11.0 WASTE DISPOSAL. N/A

12.0 METHOD PERFORMANCE. N/A

13.0 REFERENCES

13.7 High Intensity Ultrasonic Processor/Instruction Manual.

13.8 Maintenance of Zone LogBook, SWL-GA-128.

14.0 DEFINITIONS

Terminology

14.1 ON/OFF/TUNE Switch: ..... ON position energizes the power supply. OFF position de-energizes the power supply. TUNE energizes the power supply momentarily for tuning purposes.

14.2 Pilot Light: ..... Illuminates when the power supply is energized.

14.3 Continuous/Pulsed*: ..... Sets the duty cycle to continuous or pulsed mode.

14.4 % Duty Cycle Selector*:..... Sets the pulse rate when CONTINUOUS/PULSED Switch is set to pulsed. Intermittent operation inhibits heat build-up in the solution and provides more efficient processing by allowing the material to settle back under the probe tip after each burst.

14.5 Timer:..... Sets the duration of ultrasonic application (up to 15 minutes). In the HOLD position, the timing mode is uninhibited and the ultrasonic energy is applied indefinately.

14.6 Output Control: ..... Controls the amplitude of vibrations at the probe tip. Caution: When using a microtip or extender, never allow the output control setting to exceed MICROTIP LIMIT "5".

14.7 Power Monitor: ..... Indicates the percentage of ultrasonic Power delivered to the probe. (See Power Output Charts, Page 6).

14.8 Pin Connector: ..... Connects to the converter cable.

14.9 Pin Connector*: ..... Connects to the converter cable (available only with 500 watt dual output units).

14.10 Tuning Control:..... Optimizes power supply performance by tuning the power supply to the converter/probe assembly.

14.11 Fuse:..... Protects against electrical overload.

14.12 Electrical Line Cord:..... Connects the power supply to the electrical outlet.

15.0 ATTACHMENTS

- Sonicator Record

ATTACHMENT 1
Sonicator Record

SONICATOR RECORD

SONICATOR: A, B, C, E

MONTH: , 19

File Name (F:\Navy97\Sop's\Forms\Books\GA128F9.doc) SWL SOP Rev.2.1 Book ID: GA-128-ZoneSoni-3

CONTROL LIMITS: 17.5 (±3) DIV

DATE	TIME	ANALYST	WITHIN LIMITS?	CORRECTIVE ACTION* (IF NEEDED)
1			YES NO	
2			YES NO	
3			YES NO	
4			YES NO	
5			YES NO	
6			YES NO	
7			YES NO	
8			YES NO	
9			YES NO	
10			YES NO	
11			YES NO	
12			YES NO	
13			YES NO	
14			YES NO	
15			YES NO	
16			YES NO	
17			YES NO	
18			YES NO	
19			YES NO	
20			YES NO	
21			YES NO	
22			YES NO	
23			YES NO	
24			YES NO	
25			YES NO	
26			YES NO	
27			YES NO	
28			YES NO	
29			YES NO	
30			YES NO	
31			YES NO	

TYPIST: LLR

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TM

Standard Operating Procedure

General Training Of New Extraction Personnel

Document No.: SWL-OE-103

Rev No. / Date: Rev. No.: 3.0 — 05/16/00

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GENERAL TRAINING OF NEW EXTRACTION PERSONNEL

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1.0 PURPOSE AND APPLICATION

The purpose of this document is to define Extraction Laboratory general procedure. Extraction is one of the most crucial steps in quantitative organic analysis, because extraction efficiency/correctness affects every subsequent step in the analysis chain.

2.0 RESPONSIBILITIES

As defined in Section 5.0

3.0 EQUIPMENT

Not applicable.

4.0 PRECAUTIONS

4.1 Good extraction technique is tantamount to producing good analytical data. Concentrated, focused effort is crucial to minimize mistakes. Most extraction errors occur due to lack of focus. Therefore, talking must be kept at a minimum to ensure concentration on the task at hand.

4.2 Sample transfer is a critical step in sample preparation/extraction. At these junctures, the potential of accidentally switching labels or vials is high. Therefore, extreme caution must be used at these steps which include solvent exchange, GPC , and final blowdown.

5.0 PROCEDURE

5.1 Safety

5.1.1 Safety training must be completed and documented before personnel begin work in the laboratory.

5.1.2 Safety glasses and lab coats must be worn at all times by personnel when they are in the extraction laboratory.

5.1.3 See the standard operating procedure for "*Laboratory Safety Plan*", SWL-GA-111 for detailed safety information.

5.2 Paperwork

5.2.1 Paperwork is an important part of the extraction process. Thorough documentation of each extraction step, noting any unusual occurrences or method deviations, is essential to present the best service to our clients. All paperwork must be completed, thoroughly reviewed, and verified by a designated reviewer before submitting to the analyzing laboratory. The reviewer must initial and date the designated section of the extraction log.

5.2.2 All samples removed from the sample storage area must be signed out by completing the appropriate form. When the sample is returned to the sample storage area, it must be signed in by completing the next section on the same

form. NOTE: All empty containers must be logged on the appropriate form as well. See SWL-GA-110 "*Laboratory Documentation of Sample Custody*", for detailed information.

5.2.3 For samples fractions which have multiple aliquots (Logged in with a suffix identifier (eg. –a, -b, -c, etc.) that identifier must be noted on the extraction log under the comments section.

5.2.4 The correct SOP number must be entered on the extraction log before extraction is initiated. This ensures that the proper procedure is used. Always consult the Section Supervisor if unsure of which SOP to use.

5.2.5 Comments must be liberally made in the comments section of the extraction log. Detailed notes are required for any abnormalities or method deviations during extraction. The notes allow a detailed case narrative to be written without unnecessary assumptions. Solvent/Reagent lot #s or a "x" reference to the Solvent Reagent Log Book (SWL-GA-107) must be recorded.

5.2.6 Clients regularly send ampulated P.E.s to the laboratory for analysis. These P.E.s are generally spiked into a specified volume of water. For these samples, the sample amount should be noted as follows on the extraction log: the amount of P.E. spiked followed by a "/" and then the amount of water spiked (e.g., 1/1000mL would be entered for 1mL of ampulated P.E. sample spiked into 1000mL of water). Also, note in the comment section that the sample is an ampulated P.E.

5.2.7 Florisil cartridge and GPC column numbers must be entered in the appropriate section on the extraction log as well as the cleanup method used (i.e. 3620B, 3640A, etc.)

5.2.8 Spike solution numbers are entered in the appropriate sections on the extraction log.

5.3 SOP Review

5.3.1 Before proceeding with any extraction procedure, the extraction chemist must have reviewed and been trained on the appropriate SOP. Initial proficiency testing must also be completed before extraction is performed.

5.3.2 Extraction SOPs must be periodically reviewed to refresh the extraction chemist's memory. They must be consulted when any questions arise.

5.4 Weighing/Measuring

5.4.1 Targeting of sample weights is prohibited for solid samples.

5.4.2 Method-specified volumes of liquid sample must be used for extraction, even if two bottles are needed. Check to see that two bottles are available when extraction setup has begun (e.g., 2 bottles are received, both contain 850mL. The procedure calls for 1000mL. Therefore, use the entire first bottle and enough of the second bottle to bring the final volume of sample extracted to 1000mL). If

insufficient sample is available, note this in the comment section on the extraction log. Solvent rinse bottles when applicable.

5.4.3 Percent moisture and pH determinations must be completed before the sample extracts are submitted to the analyzing laboratory. If the samples are 72 hour or less turn around times, the percent moisture and pH determinations may be submitted to the analyzing laboratory no later than the evening before the due date.

5.5 Spiking

5.5.1 Surrogates are compounds that are chemically similar to the compounds of interest, but are not typically found in the environment. These compounds are used to assess extraction efficiency (testing of the extraction laboratory personnel and their technique; i.e., blanks and LCS) and matrix effect (the affect that the sample matrix may have on extraction efficiency).

5.5.2 Spike solutions must be allowed to equilibrate to room temperature before use. For a given weight of organic solvent, volume changes in response to changing temperature (volumes decrease at cooler temperatures and increase with increasing temperatures). Therefore, more surrogate or spike compound is delivered when the spike solution is cold than when it is at room temperature. This will result in inconsistent surrogate and spike recoveries and is unacceptable.

5.5.3 A verifier must be present to witness all spiking events. The verifier ensures that the correct solutions are spiked at the proper volumes and into the correct samples for each particular test code. As each sample is spiked, a check mark "✓" is made on the vessel with a Sharpie™ marker or equivalent. The check indicates that the sample has been spiked. Laboratory control spikes and matrix spikes will have two check marks indicating that both surrogate and matrix spike has been spiked into the sample.

5.5.4 No talking is allowed during spiking/verification except that which is required to perform the spiking/verification. Do not allow any personnel to interrupt during spiking/verification (i.e., proceed to a logical stopping point before acknowledging the person). Personnel must not be interrupted during the spiking/verifying process.

5.5.5 A matrix spike and matrix spike duplicate must be set up for every 20 samples per method per matrix per day. If insufficient sample is available for MS/MSD, a note must be made on the extraction log indicating such.

5.5.6 Spike solution request forms must be completed when <50% of the solution volume remains or one week prior to spike solution expiration.

5.6 pH Adjustment

It is crucial that all sample pHs be within method specifications. Therefore, the pH of all water samples must be checked for compliance to the method. If pH is not within method specifications, it must be adjusted using an acid or base solution, as appropriate. After adding the acid or base, the sample must be agitated or stirred so that adequate mixing occurs. The pH must be re-tested to ensure that it is within method specifications.

5.7 Solvent Concentration

5.7.1 K-D Concentration — See individual extraction SOPs for concentration steps using the KD Apparatus.

5.7.1.1 The temperatures for the K- D Concentration water baths are monitored and documented in a water bath logbook. (see Attachment 2).

5.7.2 Turbo-Vap Concentration

5.7.2.1 Set up glass filter funnels, with S&S # 595 filter paper, and approximately 20 grams of Sodium Sulfate. Rinse the filter paper and sodium sulfate with methylene chloride. Record the Sodium Sulfate lot # on the Extraction Log, in the comment section.

5.7.2.2 Place a glass funnel (previously prepared) in the Zymark tube.

5.7.2.3 Slowly pour the solvent from the boiling flask into the funnel.

5.7.2.4 Rinse the boiling flask with three 10mL portions of the methylene chloride. Pour these rinses through the Na_2SO_4 and collect all in the Zymark tube.

5.7.2.5 After the solvent has been transferred to the Zymark tube, remove the funnel and place the Zymark tube into the Zymark concentrator.

5.7.2.6 Concentrate the sample to a 1mL volume, rinsing the walls of the Zymark tube with 1 to 2mL of methylene chloride several times during the concentration.

NOTE: When the volume of the extract reaches the 1mL of 0.5mL mark according to the Turbovap used, a sensor will sound a long beep.

5.7.2.7 Transfer the sample to a 1.8mL Wheaton vial and put a preprinted sample ID label on the 1.8mL Wheaton vial. Sign and date the KD portion of the extraction log noting TV for turbovap.

5.7.2.8 The extract is now ready for final concentration, proceed to section 5.9.

5.8 Solvent Exchange

All exchange steps must be completed according to SOP. Exchange steps must not be skipped or shortened because portions of the extraction solvent will remain in the exchange solvent. Exchange is performed because the extraction solvent is incompatible with the instrumentation detection system. Therefore, all original solvent must be gone from the extract or severe damage to the instrument detection system may occur. See Section 5.8 for problems associated with both solvent exchange and nitrogen blowdown.

5.9 Nitrogen Blowdown

The process of nitrogen blowdown is highly technique oriented. Several problem areas should be delineated to ensure proper care is taken: 1) Low molecular weight compounds are easily "blown away" if the nitrogen stream is too vigorous; 2) if the solvent volume is reduced too low, compounds may be "blown away"; 3) Reducing the solvent to dryness will cause compounds, even heavier molecular weight compounds to be lost or to adhere to the glass vial. Addition of solvent will not bring these compounds back into solution; 4) Even subtle technique problems can be seen on the sensitive instrumentation of the analyzing laboratories; and 5) The final volume of extracts are made by comparison to marked screw cap vials.

5.10 Glassware

5.10.1 The analytical instrumentation used in organic determinations is typically sensitive from the parts-per-million range down to the parts-per-trillion range. The following example gives an idea of the magnitude of these low levels: 1 part-per-trillion is like taking a 1 second vacation after working for 31,700 years. All glassware must be scrupulously cleaned to prevent sample extract contamination since even minute amounts of contaminant will render an analysis unusable.

5.10.2 All glassware must be rinsed with the extraction solvent prior to use.

5.11 Departmental Transfer of Samples and Extracts

All samples must be signed in and out of sample receiving. No samples may be taken from Sample Receiving without first being signed out. This includes the Radiation sample receiving area. See SWL-GA-110 "*Laboratory Documentation of Sample Custody*", for detailed information.

Extracts are logged in to the analyzing laboratory when extraction and all paperwork have been completed. The extracts are taken to the analyzing laboratory with the accompanying extraction log. The sample extract episode numbers are written in the Extract Receipt Log, along with the date and time of submittal. In addition, the client name, submitting chemist's initials, whether the extracts are original or re-extracts, and comments (such as test code of extracts) are made. It is imperative that all extracts that are brought to the analyzing laboratory, whether tests or client samples, are logged into the Extract Receipt Log.

6.0 RECORDS

6.1 Records as specified in "*Personnel Training*" SWL-GA-104 shall be maintained.

6.2 See Section 5.0 for other documentation.

7.0 REFERENCES

Laboratory Documentation of Sample Custody, SWL-GA-110.

Laboratory Safety Plan, SWL-GA-111.

8.0 DEFINITIONS

Case Narrative:.....Summary of all pertinent information concerning a client's sample results, including test method, QC summary, and any unusual occurrences or method deviations.

Extraction Log: .....A log used during the extraction process, noting sample names, episode numbers, amount of sample extracted, spike amounts, and final extract volumes, etc. See Attachment 1 for example.

Laboratory Control Spike (LCS): An aliquot of spike solution containing a representative number of analytes of concern is spiked into a clean matrix (Ottawa sand or Millipore water). The sample is taken through the extraction process per SOP. This is completed to test extraction technique and ability. Analysis results of these samples allow the laboratory to ensure that extraction efficiency is acceptable.

Matrix Effect:.....When a sample matrix causes interferences with target analyte identification or with passing quality control checks.

Matrix Spike/Matrix Spike Duplicate (MS/MSD): An aliquot of spike solution containing a representative number of target analytes is spiked into a client's sample. The sample is taken through the extraction process from the particular sample matrix. Analysis results of these samples allow the client to make informed decisions on data quality.

Nitrogen Blowdown:.....A concentration technique used to reduce the volume of organic extraction solvents.

P.E.:.....Performance evaluation sample used to assess the laboratory's performance.

SOP:.....Standard operating procedure.

Surrogate:.....Surrogates are compounds that are chemically similar to the compounds of interest, but are not typically found in the environment. These compounds are used to assess extraction efficiency (testing of the extraction laboratory personnel and their technique; i.e., blanks and LCS) and matrix effect (the affect that the sample matrix may have on extraction efficiency).

Target analyte: .....A chemical compound that is tested for by a particular method.

Targeting Weights:.....Weighing of an exact weight of sample (e.g., method requires 30.0 grams of sample to be extracted; the analyst weights 30.0 grams by adding or subtracting small amounts of sample).

9.0 ATTACHMENTS

Sample Extraction Log

ATTACHMENT 1

Sample Extraction Log

☐ RUSH ☐ REEXTRACT ☐ TCLP ☐ [illegible]N

SAMPLE EXTRACTION LOG

SWLO
SOUTHWEST LABORATORY OF OKLAHOMA, INC

BATCH: 970617WE

EXTRACT DATE: 1997-06-17 11:57

	[illegible]	PCB	OTHER
CONT EX			
SEP-F		XXXXX	
SHAKER			
SONC.			
SOXHLET			

SAMPLE ID (EPISODE #)	CLIENT CASE	DATE RECEIVED CLIENT SAMPLE ID	MATRIX LEVEL	SAMPLE pH	% MOISTURE %	AMOUNT EXTRACTED g or ml	EXTRACTED VOLUME ml	GPC VOLUME ml	GPC ELUTE VOL ml	FLORISIL VOL CONC ml	FLORISIL FIN VOL CONC ml	VOL SV (p) ml	FIN PROCESS VOLUME ml	SCREEN Y/N	MS SV (p) µL	SURR SV (p) µL	BASE pH CONFIRMATION	ACID pH CONFIRMATION	TDS < 10000 Y/N
BL0617WE			W			1000	250					10.0	1.0	N		1000			N
BB0617WE			W			1000	250					10.0	1.0	N		1000			N
LC0617WE			W			1000	250					10.0	1.0	N	1000	1000			N
LD0617WE			W			1000	250					10.0	1.0	N	1000	1000			N
29684.01 GC817	AATS-E 25501	1997-06-12 15:15 X6236 (CASE#25501)	W			1000	250					10.0	1.0	N		1000	<2	<2	N
29684.02 GC817	AATS-E 25501	1997-06-12 15:15 X6237 (CASE#25501)	W			1000	250					10.0	1.0	N		1000	<2	<2	N
29685.01 GC817	XXXXXXXX XXXXXXXX	XXXXXXXXXXXXXXXX XXXXXXXXXXXXXXXXXXXX	W			1000	250					10.0	1.0	N		1000	<2	<2	N
29685.02 GC817	XXXXXXXX XXXXXXXX	XXXXXXXXXXXXXXXX XXXXXXXXXXXXXXXXXXXX	W			1000	250					10.0	1.0	N		1000	<2	<2	N

INITIALS

WEIGHING:	BL	EXTRACTION:	BL
CLEANUP:		KD:	BL
BLOWDOWN:	BL	TV:	
SURROG. SPIKER:	BL	VERIFIER:	
MATRIX SPIKER:	BL	VERIFIER:	
SUBMITTED BY:	BL	REVIEWED BY:	

ID NUMBERS

FLORISIL CARTRIDGE #:

GPC COLUMN #:

SURROGATES ID CONC #:

MATRIX SPIKE SOLID/CONC #(s):

METHYLENE CHLORIDE LOT #:

SODIUM SULFATE LOT #:

COMMENTS

ATTACHMENT 2

Water Bath Temperature Log

SWOK / AATS ORGANIC EXTRACTION LABORATORY **BATH #__**

WATER BATH TEMPERATURE LOG

WATER BATH TEMP LOG (103-ATT2) / SWL-OE-103, Rev. 3.0 / BOOK # OE-103-WBTEMP.XL1

CONTROL LIMITS:
WATER EXTRACTS: 60-65 Degrees C. / SOIL EXTRACTS: 65-75 Degrees C

DATE	TIME	TEMP. (Degrees C.)	METHOD(S):	MATRIX	ANALYST

Southwest Laboratory of Oklahoma, Inc. / American Analytical & Technical Services, Inc.
1700 W. Albany * Broken Arrow, Oklahoma 74012 * Office (918)251-2858 * Fax (918)251-2599

SOUTHWEST LABORATORY OF OKLAHOMA, INC. *and*
AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.

TM

Standard Operating Procedure

Extraction Sheet Review

Document No.: SWL-OE-104

Rev No. / Date: Rev. No.: 1.0 — 07/21/99

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APPROVALS	DATES
Harry M. Boyd Procedure Prepared By	7-23-99 Date
Harry M. Boyd Program Manager or Section Supervisor	7-23-99 Date
Chuck Hoover Laboratory Safety Officer	7/25/99 Date
Chuck Hoover Laboratory QA/QC Officer	7/25/99 Date

(Effective Date is 5 calendar days after the last signature above - QA/QC Officer)

Document Status

[ ] Controlled DC No. _____ Issued to: _____ Date: _____ QA: _____

[✓] Official Copy OC No.: 2472 Issued to: Linda Kassonovich Date: 05/01/01 QA: [illegible]

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EXTRACTION SHEET REVIEW

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1.0 SCOPE AND APPLICATION

1.1 The purpose of this Standard Operating Procedure is to provide guidelines for extraction sheet data review. It will provide specific instructions to ensure the review process is performed with consistency.

1.2 The extraction department supervisor will be responsible for the final review of the extraction sheets. All extraction technicians are responsible for initial review of the extraction sheets, which they generate.

2.0 METHOD SUMMARY

Not applicable.

3.0 HEALTH AND SAFETY

Not applicable.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

Not applicable.

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

Not applicable.

6.0 EQUIPMENT/APPARATUS

- 486 Personal Computer (for generation of forms)

7.0 REAGENTS

Not applicable.

8.0 PROCEDURE

8.1 Located at the top, center of the Sample Extraction Log are the batch and extraction dates. The Batch date consists of the date that the samples were extracted (starting with the year, month, and day), and ends with a matrix identifier such as "W" for water or "S" for soil, followed by a sequential letter of the alphabet: i.e. 980716WF. The reviewer must make sure the Batch corresponds to the extract date.

8.2 The far left-hand column of the extraction sheet is labeled "Sample I.D. (Episode #)". The first listing in the column is the blank and back-up blank, then the LCS and LCSD if applicable. Listed next are the samples which are identified by a 5-digit episode number, and period mark, and the sample number, i.e. xxxxx.01, xxxxx.02, xxxxx.03, etc (where xxxxx is the episode number for these samples). The reviewer is responsible for assuring that all samples are accounted for.

Located directly below each sample number is the test-code i.e. MS517, which represents the test required to be performed on the sample. The reviewer should make sure these codes are consistent and correspond to the project status sheet.

8.3 The next column to the right is titled Client/Case, and identifies the client for which these samples are being analyzed.

8.4 The next column over is the Date Received and Client Sample ID column. The date received is the date that the samples were logged into the laboratory. The client sample ID is the client's identifier used to distinguish one sample from another. The reviewer must assure that these 2 items are present for each sample.

8.5 The next 17 columns over are pertinent information about the sample and the extraction procedure such as pH, %moisture, amount extracted, etc. The reviewer is responsible for making sure that all of this information is filled out completely. If it is incomplete then it is the responsibility of the reviewer to bring it to the attention of the extraction technician and get the complete information.

8.6 At the bottom of the page at the far left is a box labeled "Initials", and spaces for Weighing; Cleanup; Blowdown; Surrogate Spiker; Matrix Spiker; Submitted By; Extraction; KD; TV; Verifier; Verifier; Reviewed By; Florisil. It is the responsibility of the reviewer to make sure that these sections are initialed by the extraction technician and signed and dated by the extractions supervisor as the initial reviewer.

8.7 The next box over to the right is labeled ID numbers. This space is intended for the extractions technician to identify the materials used in the extraction procedure such as Florisil Cartridge #, GPC Column #, Surrogate ID Conc. #, Matrix Spike Solid/conc. #(s), and SOP-Method #: SWL-OE-___ Rev.___.

It is the responsibility of the reviewer to make sure that all of this information which is applicable to the samples is filled out completely. If it is not complete then the reviewer must bring it to the attention of the extraction technician and get the complete information.

8.8 The far right box is for comments for the technician to note any unusual or out of the ordinary occurrences that may have taken place during the extraction procedure that may affect the analysis results in some way. The reviewer is responsible for reading all comments to be aware of situations that have occurred that may effect the analysis results.

9.0 CALCULATIONS

Not applicable.

10.0 QUALITY ASSURANCE/QUALITY CONTROL

Not applicable.

11.0 WASTE DISPOSAL

Not applicable.

12.0 METHOD PERFORMANCE

Not applicable.

13.0 REFERENCES

Not applicable.

14.0 DEFINITIONS

Not applicable.

15.0 ATTACHMENTS

Attachment 1: Sample Extraction Log

ATTACHMENT 1
Sample Extraction Log

SAMPLE EXTRACTION LOG

SWLO
SOUTHWEST LABORATORY OF OKLAHOMA, INC.

☐ RUSH ☐ REEXTRACT ☐ TCLP ☐ DIOXIN

9/24

	SV	PCB	OTHER
CONT EX. (3520B)	XXXXX		
SEP-F (3510B)			
SHAKER			
SONC. (3550)			
SOXHLET			

BATCH: 980910WE

EXTRACT DATE: 1998-09-10 15:30

001560
Page: 1 of 1

SAMPLE ID (EPISODE #)	CLIENT / CASE	DATE RECEIVED / CLIENT SAMPLE ID	MATRIX LEVEL	SAMPLE pH	% MOISTURE (%)	DRY WEIGHT (g)	AMOUNT EXTRACTED (g or ml)	EXTRACTED VOLUME (ml)	GPC VOLUME (ml)	GPC ELUTE VOL (ml)	FLORISIL VOL CONC (ml)	FLORISIL FIN VOL CONC (ml)	VOL SV P (ml)	FIN PROCESS VOLUME (ml)	SCREEN Y/N (Y/N)	MS SV P (µL)	SURR SV P (µL)	BASE pH CONFIRMATION	ACID pH CONFIRMATION
BL0910WE			W/L				1000	450	N/A	N/A	N/A	N/A	10.0	0.5	N		500		<2
BB0910WE			W/L				1000	250	N/A	N/A	N/A	N/A	10.0	0.5	N		500		<2
35534.01 MS517	AATS-E	09/10/1998 10:17 FFR71 (CASE#26465)	W/L	5.81			1000	250	N/A	N/A	N/A	N/A	10.0	0.5	N		500		<2
35534.02 MS517	AATS-E	09/10/1998 10:17 FFS51 (CASE#26465)	W/L	7.47			1000	250	N/A	N/A	N/A	N/A	10.0	0.5	N		500		<2

INITIALS

WEIGHING:	TM	EXTRACTION:	TM	FLORISIL:	
CLEANUP:		KD:			
BLOWDOWN:	PHP	TV:	TM		
SURROG. SPIKER:	OES	VERIFIER:	TM		
MATRIX SPIKER:		VERIFIER:			
SUBMITTED BY:	PHP	REVIEWED BY:	9/14/98		

ID NUMBERS

FLORISIL CARTRIDGE #:

GPC COLUMN #:

SURROGATES ID CONC #: 1-073-14

MATRIX SPIKE SOLID/CONC #(s):

SOP - METHOD # : SWL-OE- 501 Rev. 4.2

COMMENTS

SOUTHWEST LABORATORY OF OKLAHOMA, INC. *and*
AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.

TM

Standard Operating Procedure

Extraction of Polynuclear Aromatic Hydrocarbons, Method 8310

Document No.: SWL-OE-204

Rev No. / Date: Rev. No.: 1.0 — 10/19/00

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APPROVALS	DATES
Procedure Prepared By	10/20/2000 Date
Program Manager or Section Supervisor	10-24-00 Date
Laboratory Safety Officer	10/24/2000 Date
Laboratory QA/QC Officer	10/24/00 Date

(Effective Date is 5 calendar days after the last signature above - QA/QC Officer)

Document Status

[ ] Controlled DC No. _____ Issued to: _____ Date: _____ QA: _____

[✓] Official Copy OC No.: 2472 Issued to: Linda Karsonovich Date: 05/01/01 QA: _____

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EXTRACTION OF POLYNUCLEAR AROMATIC HYDROCARBONS, METHOD 8310

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Typist: LLR

1.0 SCOPE AND APPLICATION

1.1 This SOP (SWL-OE-204) is used for the extraction (Methods 3510C and 3520C) of polynuclear aromatic hydrocarbons for all types of matrices, such as ground and waste waters, soils (Method 3550B), and all other types of solid waste matrices (Method 3550B also), with methylene chloride solvent. This extraction procedure is used for the Southwest Laboratory internal test code GC420 and when applicable GC499, as a non-standard code.

1.2 This procedure contains no deviations from the methods listed above.

2.0 METHOD SUMMARY

2.1 A measure volume of sample is extracted with methylene chloride usually 1 liter. For water samples, the pH is adjusted if needed to two. The methylene chloride extract is dried and concentrated to a volume of 1.0mL. Chromatographic conditions are described which permit the separation and measurement of the parameters in the extract. Qualitative identification is performed using the retention time and the relative abundance of three characteristic ions. Quantitative analysis is performed using internal standard techniques with a single characteristic ion.

2.2 A measured weight, (normally 30 grams for low level organic or 1 gram for medium/high level organics) of solid or non-aqueous liquid, is mixed with a drying agent and extracted with a methylene chloride/acetone mixture by sonication. This extract is concentrated to a final volume to 0.5mL.

3.0 HEALTH AND SAFETY

3.1 Caution should be taken when extracting all samples. Most samples are of unknown origin and should be considered hazardous. Many of the spiking solutions used, along with chlorinated solvents are considered carcinogenic. Concentrated acids and bases, if not handled properly, can cause severe burns.

3.2 Gloves should be used for the extraction of all samples and preparation of all acid and base solutions. Wear personal protective equipment (PPE) and goggles or face shield when warranted. See SOP SWL-GA-111, "*Laboratory Safety Plan.*"

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

4.1 Extraction holding times for samples are as follows: 5 days from the date of VTSR aqueous samples and 10 days from the date of VTSR for soil/solid samples.

4.2 No sample preservation is necessary for organic semivolatiles, however samples and final extracts must be kept at 4° Celsius ±2 degrees.

4.3 All containers should be glass jars with Teflon lids. A minimum of one liter is required for aqueous samples. A minimum of 30 grams is required for soil samples, (125mL glass jar).

NOTE: Amber bottles should be used for aqueous samples.

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

5.1 Extreme care must be taken when extracting samples in an effort to minimize laboratory contamination. This contamination can come from unclean glassware, gloves, and any other apparatus being used in the extraction area. The use of many types of protective equipment, especially gloves, contain high levels of phthlates. Many of these phthlates interfere with the analysis of target compounds. Therefore, extreme caution must be taken. One must refrain from touching the inside of any container, any solvent, or any apparatus when wearing gloves.

5.2 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or cause interference's to sample analysis. All these materials must be demonstrated to be free from interference under the conditions of the analysis by analyzing method blanks. Specific selection of reagents and purification of solvents by distillation in all-glass system may be required.

5.3 Phthalate ester contamination is commonly found in the laboratory. Plastics, in particular, must be avoided because phthalates are commonly used as plasticizers and are easily extracted from plastic materials. Serious phthalate contamination may result at any time if consistent quality control is not practiced.

5.4 Glassware contamination resulting in analyte degradation: Soap residue on glassware may cause degradation of certain analytes. Specifically, aldrin, heptachlor, and most organophosphorous pesticides will degrade in this situation. This problem is especially pronounced with glassware that may be difficult to rinse (e.g., 500mL K-D flask, Snyder columns, and condenser tubes). These items should be hand-rinsed very carefully to avoid this problem (see SOP SWL-GA-121, "*Glassware and Labware Cleaning.*")

6.0 EQUIPMENT/APPARATUS

6.1 General Extraction Equipment and Apparatus

6.1.1 400mL Pyrex beakers

6.1.2 pH meter, (Orion 420A), or equivalent

6.1.3 pH-paper, narrow range (1-6) and (7-11)

6.1.4 Short stem glass funnels: top ID 100mm, stem 100mm, (Pyrex and Kimax)

6.1.5 Filter paper, (Schleicher & Schuell #595), or equivalent

6.1.6 Kuderna Danish concentrator apparatus (500mL)

6.1.7 3 ball Snyder columns

6.1.8 10mL concentrator tubes

6.1.9 5 ¾ or 9 inch disposable pipettes

6.1.10 1.8mL extract vials (Wheaton or equivalent)

6.1.11 20mL scintillation vials (Wheaton or equivalent)

6.1.12 PFE-Latex Gloves, (Safeskin or equivalent)

6.1.13 Temperature controlled hot water bath

6.1.14 Glass stirring rods

6.2 Soil Extraction Equipment and Apparatus

6.2.1 Stainless steel spatulas

6.2.2 Sonicators (Vibra Cell model VC602) or equivalent, with ¾ inch probe or 4-post micro probe.

6.2.3 GPC – Low Pressure – ABC 1002A or ABC 1002B – or equivalent

6.2.4 GPC – High Pressure – Zymark (Benchmate) dual column, Marathon single column, or equivalent.

6.2.5 Aluminum weighting Pans (70 x 16mm)

6.2.6 Electronic Balance (OHAUS Galaxie 400 or equivalent)

6.2.7 10mL PP+PE sterile disposable syringes

6.2.8 Acrodics, 0.45 micron, (Gelman or equivalent)

6.2.9 Automated Shaker

6.3 Water Extraction Equipment and Apparatus

6.3.1 2000mL Separatory Funnel

6.3.2 1000mL Graduated cylinders

6.3.3 500mL boiling flasks

6.3.4 Liquid-Liquid extractors capable or holding 1300mL of liquid

6.3.5 Boiling Beads (Hengar Granules plain and micro) or equivalent

6.3.6 Automated electrical heating panel to control the 18-hour extraction setting for liquid-liquid extractors

6.3.7 4 ounces specimen containers (polypropylene with polypropylene caps)

7.0 REAGENTS

7.1 Reagents

7.1.1 Reagent water, ASTM type II, as described in the water quality SOP

7.1.2 Sulfuric Acid (Fisher certified A.C.S. PLUS) or equivalent

7.1.3 Sodium Hydroxide pellets (Fisher certified A.C.S.) or equivalent

7.1.4 Sodium Sulfate, Anhydrous crystals (JOST low organic A.C.S.) or equivalent

7.2 Solvents

7.2.1 Methanol (Fisher A.C.S. grade spectra analyzed) or equivalent

7.2.2 Methylene Chloride (Fisher OPTIMA), or equivalent

7.2.3 Acetone (Fisher GC Resolv), or equivalent

8.0 PROCEDURE

8.1 Preliminary Procedures

8.1.1 Retrieve water samples from sample storage, and fill out sample internal chain of custody records before removing samples from sample storage area

8.1.2 Generate water extraction log and review and complete pertinent information: Client, Date received, Extraction Date, SWLO Sample ID, client sample ID, Extraction type, matrix, etc.

8.1.3 Prepare preprinted extraction labels for all samples and QC (each sample should have 4 preprinted labels to be used during extraction and final blow-down.

8.2 Extraction of PAH from Water

8.2.1 Pre-rinse all glassware and apparatus with the extraction solvent to be used.

8.2.1.1 Shake sample bottle well

8.2.1.2 Make sure enough sample is retained for pH determination (approximately 20mls).

8.2.1.3 When a continuous extractor is used, 250mls of methylene chloride is added to the continuous extractor and 200mls to the boiling flask.

8.2.1.4 A one-liter portion of sample water is measured volumetrically and poured into the continuous extractor. Record the volume on the extraction data sheet.

NOTE: After all samples are weighted out, all remaining sample must be returned to the sample storage area, and the internal chain of custody records must be filled to indicate that the remaining sample has been returned. If the entire sample was used for the extraction, then the Pitched Bottle Log must be filled out. This log is with the internal chain of custody records.

8.2.1.5 To water samples are added 0.5ml of surrogate spiking solution and to the matrix and matrix duplicate samples are added 0.5mls of matrix standard spiking solution. Record amount and ID of spiking solution on extraction sheet when added. Spiking verifier must initial.

NOTE: Spiking and Verifying all samples. Each spiker and verifier must first back-check each extraction log with the extraction sample log-in record in order to ensure that the specified samples for MS/MSD, etc. are correct. Also, if MS/MSD are given by the extraction lab, one sample must be used for both the MS/MSD (e.g. 37305.01ms, 37305.01msd). The verifier needs to verify that the correct solutions are being spiked at the proper volumes and are spiked into the correct samples. As each sample is spiked, a check mark (✓) is put on the vessel to indicate that sample has been spiked.

Lab control spikes and matrix spikes will have two "✓" marks. One for surrogate and one for matrix spike.

8.2.1.6 All samples including blanks are pH adjusted to neutral checking the pH with pH paper. pH verification is noted and initialed on extraction data sheet.

8.2.2 Sample Extraction

8.2.2.1 Assemble the boiling flask, the liquid-liquid extractor and the condenser on the automated 18-hour liquid-liquid extraction apparatus.

8.2.2.2 Open stopcock on the liquid-liquid extractor so that the extractor will work properly. Solvent will start to siphon as distillation starts.

8.2.2.3 Check the cool-flow water re-circulator, if need turn on and inspect to ensure re-circulator is working.

8.2.2.4 Turn on the 18-hour automated heating mantles and let samples extract.

8.2.2.5 After samples have started extracting, check each liquid-liquid extractor to ensure that all joints are connected properly and extraction solvent is not leaking at any joint.

8.2.2.6 After the completion of the 18-hour extraction period the heating mantles will automatically shut off.

8.2.2.7 After the boiling flasks have cooled down, shut off the stopcock on the liquid-liquid extractors, and remove the water condensers.

8.2.2.8 Check the pH of extracted water to verify that pH is still neutral, if not, then readjust pH and repeat steps (8.2.2.1 – 8.2.2.7.)

8.2.2.9 When the extraction is completed the pH of the sample is re-checked to verify it is neutral. This verification is noted on the extraction data sheet.

8.2.2.10 Upon completion of the extraction, the extracted water and remaining methylene chloride in the liquid-liquid extractor is transferred to a separation vessel so that the solvent can be separated from the water.

8.2.2.11 The waste solvent is disposed as per the SOP SWL-GA-114 "*Hazardous Waste Management Plan*", (see Section 13.0 "References" for additional information.)

8.2.2.12 The extracted water is disposed in the normal manner.

8.2.2.13 The extract in the boiling flask is dried, by using sodium sulfate, prior to concentration by the KD method.

8.2.3 Initial Extract Concentration

8.2.3.1 KD method

8.2.3.2 Set up glass filter funnels, with S&S #595 filter paper, and approximately 20 grams of Sodium Sulfate. Rinse the filter paper and sodium sulfate with methylene chloride. Record the Sodium Sulfate lot # on the Extraction Log, in the comment section.

8.2.3.3 Attach a concentrator tube to a KD flask and rinse both with methylene chloride.

8.2.3.4 Place a glass funnel containing sodium sulfate (previously prepared) above the KD assembly.

8.2.3.5 Slowly pour the solvent from the boiling flask into the funnel.

8.2.3.6 Rinse the boiling flask with approximately 10mL of the methylene chloride. Repeat this process two additional times. Each time pouring the rinses through the Na_2SO_4 and collecting in the KD.

8.2.3.7 After the solvent has been transferred to the KD flask, remove the funnel. Add two clean boiling chips to the KD.

8.2.3.8 Attach a three-ball Snyder column to the KD. Pre-wet the column with approximately 1mL of methylene chloride.

8.2.3.9 Place the KD assembly in a hot water bath (not above 65°C) so that the entire lower rounded surface of the KD assembly is immersed in the water. The position of the KD assembly in the water bath and the water bath temperature should be such that the concentration is completed in 45-60 minutes. The balls of the Snyder column should actively chatter and the chambers should not flood with condensed solvent.

8.2.3.10 When the apparent volume of liquid reaches approximately 10mL, add 1.0mL cyclohexal at the top of the Snyder column, and remove from the water bath as soon as possible. Rinse the Snyder column with approximately 1mL of methylene chloride, and allow the apparatus to drain and cool for at least 10 minutes. If the sample will not concentrate to a 10mL volume, consult your supervisor for instructions. Sign and date the KD portion of the extraction log.

8.2.3.11 Remove the Snyder column.

8.2.3.12 Transfer the contents from the KD assembly to a 20mL scintillation vial.

8.2.3.13 Rinse KD assembly three separate times, with 1-2mL of methylene chloride, and pour each rinse into the scintillation vial.

8.2.3.14 Place a preprinted sample ID label on scintillation vial and proceed to Section 8.2.3.15.

8.2.3.15 Gently blow down the extract to 0.5mL and add 1.0mL cyclohexane and reduce the volume again to 0.5mL. Add cyclohexane and adjust the volume to exactly 2.0mLs. The extracted is now ready for silica gel chromatography. After the silica gel cleanup process, add 0.5mL acetonitrile tot he extract and continue the blowdown process to 0.5mL. Add 1.0mL acetonitrile and again reduce the 0.5mL. Rinse and transfer the extract to a pre-rinsed 12mL vial not to exceed 1.0mL. Add exactly 1.0mL Millipore water and bring to a final volume of 2.0mLs.

8.3 Extraction of PNA Fractions from Soil/Sediments — Low Level

8.3.1 Retrieve soil samples from sample storage, and fill out sample internal chain of custody records before removing samples from sample

8.3.2 Generate soil extraction log and review and complete pertinent information: Client, Date received, Extraction Date, SWLO Sample ID, client sample ID, Extraction type, matrix, etc.

8.3.3 Prepare preprinted extraction labels for all samples and QC, each sample should have four pre-printed labels to be used during extraction and final blowdown.

8.3.4 Retrieve sufficient amount of 400mL beakers, one for each sample + QC, and place a preprinted sample ID label on each beaker.

8.3.5 Homogenize sample before taking sub-samples. This is done, by carefully removing any large stones, sticks, and then decanting any water at the top of the sample before mixing. Then mix the sample in the sample container by using a stainless steel spatula.

8.3.6 Make pH determination of each sample (see Section 8.5.3 for procedure,) if pH is <5 or >9 pH units, inform the supervisor of the pH on these samples. Record pH on the Extraction Log for each sample.

8.3.7 Determine the moisture content of each sample (see Section 8.6 for procedure.) Record percent moisture on the Extraction Log for each sample.

8.3.8 Calibrate the balance before weighing samples if balance was not previously calibrated for the day. Check Zone Log Book on procedure for calibration balance.

8.3.9 Weigh 30 grams of the sample to the nearest 0.1 grams into a 400mL beaker. Record the weight on the Extraction Log for each sample plus QC. For blanks and laboratory control spikes (when needed for non-EPA CLP clients,) pre-extracted white sand will be used for the 30 grams sample weight.

8.3.10 Add approximately 60 grams of anhydrous sodium sulfate, to the 400mL beaker and mix well with a glass rod or stainless steel spatula.

Note: If samples are not free flowing, add additional sodium sulfate. Record the sodium sulfate Lot # on the Extractions Log, in the "Comment" section.

8.3.11 Continue doing 8.3.9 and 8.3.10 for each sample plus QC being extracted.

Note: After all samples are weighted out, all remaining sample must be returned to the sample storage area, and the internal chain of custody records must be filled to indicate that the remaining sample has been returned. If the entire sample was used for the extraction, then the Pitched Bottle Log must be filled out. This log is with the internal chain of custody records.

8.3.12 Surrogate Spike Addition — To each sample plus QC add 750uL of surrogate spiking solution. Check to verify that you have the correct surrogate spiking solution, for the test code being performed, and that the surrogate spiking solution is not expired. Record surrogate spiking solution ID#, the spiking of each sample, and sample spike verification (second person) on Extraction Log.

To each beaker, add 100mL of methylene chloride. Record the methylene chloride Lot # and the Acetone Lot # on the Extraction Log, in the comments section.

8.3.13 Matrix Spike (MS) Addition — To each Matrix Spike Sample (MS) or duplicate (MSD), add 750uL of MS spiking solution. Check to verify that you have the correct MS spiking solution for the test code being performed and that the MS spiking solution is not expired. Record the MS spiking solution ID #, the spiking of MS samples, and sample spike verification (second person) on the Extraction Log. <u>The matrix spike spiking solution is prepared and tested by the HPLC Department prior to use. When the solution is approximately 50 percent used up or expired, a new solution must be requested from the Semivolatile Department.</u>

Note: The verifier needs to verify that the correct solutions are being spiked at the proper volumes and are spiked into the correct samples. As each sample is spiked a "✓" is labeled on the vessel to indicate that sample has been spiked.

Lab control spikes and matrix spikes will have two "✓" marks; one for surrogate and one for matrix spike.

8.3.14 Sample Sonication

8.3.14.1 Set up glass funnels, with S&S # 595 filter paper, and approximately 10 to 20 grams of Sodium Sulfate, in 8-port soil extraction funnel holder (top level.)

8.3.14.2 Set up Kuderna Danish concentrator assembly with boiling chips in the 8-port soil funnel holder (lower level.)

8.3.14.3 Install preprinted sample ID labels on Kuderna Danish concentrator.

8.3.14.4 Sonicate the samples using the ¾ inch probe held about ½ inch below the liquid surface but ½ inch above the sediment level.

8.3.14.5 Use full power and a 50% pulse for 3 minutes.

8.3.14.6 Stir the extract with the stainless steel spatula, then decant and filter the extract through a filter paper in a glass funnel, collecting the extract in the KD.

Note: Extreme care must be taken to ensure that the correct sample is put into properly labeled KD, as not to switch samples.

8.3.14.7 Add another 100mL of the methylene chloride to the 400mL beakers containing the samples, stir the sample with the stainless steels spatula and repeat sections 8.3.14.4 – 8.3.14.6. Repeat this section twice.

8.3.14.8 Rinse beaker and filtered funnel with approximately 50mL of methylene chloride.

8.3.14.9 Check Sodium Sulfate. If the sodium sulfate has hardened, then the methylene chloride extract must be re-dried using new sodium sulfate. This must be done before the sample extract is concentrated. When the extract is deemed dry, then proceed to Section 8.3.14.10.

8.3.14.10 Initial Extract Concentration

- Attach a three-ball Snyder column to the KD. Pre-wet the column with approximately 1mL of methylene chloride.
- Place the KD assembly in a hot water bath (not to exceed 65°C)so that the entire lower rounded surface of the KD assembly is immersed in the water. the position of the KD assembly in the water bath and the water bath temperature should be such that the concentration is completed in 45-60 minutes. The balls of the Snyder column should actively chatter and the chambers should not flood with condensed solvent.
- When the apparent volume of liquid reaches approximately 10mL, add 1.0mL cyclohexane at the top of the Snyder column and remove from the water bath as soon as possible. Rinse the Snyder column with approximately 1mL of methylene chloride, and allow the apparatus to drain and cool for at least 10 minutes. If the sample will not concentrate to a 10mL volume, consult your supervisor for instructions. Sign and date the KD portion of the Extraction Log.
- Remove the Snyder column.

- Transfer the contents from the KD assembly to a 20mL scintillation vial.
- Rinse KD assembly three separate times, with 1-2mL of methylene chloride, and pour each rinse into the scintillation vial.
- Place a pre-printed sample ID label on scintillation vial.
- Place the scintillation vial on the nitrogen blow-down apparatus. The flow of nitrogen should be such that it causes a slight ripple across the top of the sample without causing splashing of the sample. Concentrate the sample to 10mL.
- Filter each sample extract plus QC through an Acrodics, 0.45 micron, (Gelman or equivalent,) using 10mL PP+PE sterile disposable syringes and place filtered extract back into a labeled 20mL scintillation vial.

8.3.14.11 Gently blow down the extract to 0.5mL, and add 1.0mL Cyclohexane and reduce the volume again to 0.5mL. Add cyclohexane and adjust the volume to exactly 2.0mLs. The extract is now ready for Silica gel chromatography. After the silica gel cleanup process, add 0.5mL acetonitrile to the extract and continue the blow down process to 0.5mL. Add 1.0mL acetonitrile and again reduce the 0.5. Rinse and transfer the extract to a pre-rinsed 12mL vial not to exceed 1.5mL. Add exactly 1.5mL Millipore water, and bring to a final volume of 3.0mLs.

8.3.14.12 Silica Gel Chromatography

The soil/water extract ready for chromatography must be at 2.0mL in cyclohexane. Add 10g activated silica gel to a 10mm ID column, and tap gently. Add 1-2cm of pre-backed anhydrous sodium sulfate to the top of the silica gel and tap gently. Add 20mLs methylene chloride to the column and eluate discarding the solvent. Prior to the exposure of the sodium sulfate layer, add 40mL pentane and discard.

Note: All elution rates should be approximately 2mL/minute, and the column must not go dry any time during the process except the last stage.

Add the 2mL sample to the column and rinse the extract vial with 1-2mL cyclohexane. Add 25mL pentane and discard the eluate. Add 25mL methylene chloride/pentane (2:3) (v/v) and collect the eluate. Blow down, exchange the methylene chloride/pentane solutions to acetonitrile, and submit for analysis (soils 3.0mL, water 2.0mL.)

8.4 pH Determination

8.4.1 The pH meter is to be calibrated before use and calibration data recorded in the pH meter logbook.

8.4.2 Water Samples

8.4.2.1 Shake the sample, and transfer approximately 20mL to a clean scintillation vial.

8.4.2.2 Place the pH probe into the liquid and allow the meter to obtain a stable reading.

8.4.2.3 Record the pH reading on the sample data and on the extraction log.

8.4.3 Soil Samples

8.4.3.1 Homogenize the sample.

8.4.3.2 Weigh approximately 30 grams of sample into a clean polypropylene container.

8.4.3.3 Add 30mL of deionized water to the sample.

8.4.3.4 Mix sample and water with a stainless steel spatula.

8.4.3.5 Cover the polypropylene container with a polyethylene cap.

8.4.3.6 Allow the sample to shake for one hour on an automatic shaker.

8.4.3.7 After 1 hour, place the pH probe into the liquid and allow the meter to obtain a stable reading.

8.4.3.8 Record the pH reading on the sample data sheet and on the extraction log.

8.5 Moisture Determination

8.5.1 Use Southwest Laboratories Automated Percent Moisture Program.

8.5.2 Enter in Southwest Laboratory ID (episode number) for sample.

8.5.3 Label (using a marker) the aluminum weighting pan (70 x 16mm), with same ID as in episode.

8.5.4 Tare the aluminum weighting pan (70 x 16mm) on the balance.

8.5.5 Weigh 5-10 grams of the sediment into the aluminum weighting pan (70 x 16mm.)

8.5.6 Place samples into a 103 ±3 degrees Celsius drying oven.

8.5.7 Dry samples overnight.

8.5.8 Remove samples from oven and allow the samples to cool at room temperature before weighing.

8.5.9 Re-weigh the aluminum pans (containing the dried sample) on the balance using the automated percent moisture program.

8.5.10 Print out the percent moisture reports from the automated program and store in a 3-ring binder.

8.5.11 Record the percent moisture on the Extraction Log.

9.0 CALCULATIONS

Percent Moisture Calculation:

$$\text{Percent Moisture} = \frac{(\text{wet weight of sample} - \text{dry weight of sample})}{(\text{wet weight of sample})} \times 100$$

10.0 QUALITY ASSURANCE/QUALITY CONTROL

The HPLC department monitors Quality Assurance/Quality Control. This is accomplished by reviewing the extraction recoveries on the matrix spikes and surrogates.

11.0 WASTE DISPOSAL/POLLUTION PREVENTION

All organic extraction waste is to be disposed in accordance with SOP SWL-GA-114, "*Hazardous Waste Management Plan.*"

12.0 METHOD PERFORMANCE

The HPLC department monitors method performance. This is accomplished by reviewing the extraction recoveries on the matrix spikes and surrogates.

13.0 REFERENCES

United States Environmental Protection Agency, "*Determination of Polycyclic Aromatic Hydrocarbons in Ground Water an Wastes,*" SW-846 Method 8310, 1986.

Unites States Environmental Protection Agency, "*Determinative Chromatographic Separations,*" SW-846 Method 8300B, Revision 2, 1996.

14.0 DEFINITIONS

cont. Ex: .....Abbreviation for "continuous liquid-liquid extractor" used in the extraction of aqueous water samples.

Liquid-liquid: .....Abbreviation for "continuous liquid-liquid extractor" used in the extraction of aqueous water samples.

Aqueous Liquid:...A liquid, which is soluble in water.

PPE: .....Personnel Protective Equipment

Degradation:.....The breaking down of a compound into different compounds.

QC:.....Quality Control, used in this SOP to indicate types of samples such as extraction blanks, laboratory control spikes, and matrix spikes.

LCS:.....Laboratory Control Spike

LCSD:.....Laboratory Control Spike Duplicate

MS:.....Matrix Spike

MSD:.....Matrix Spike Duplicate

(second person):...The person doing the spike verification for sample spiking.

KD:.....Kuderna Danish

mL:.....Millimeters

gm:.....Grams

ID:.....Identification

#:.....Number

1:1.....Represents a 50 percent volume to 50 percent volume ratio

S&S # 595:.....Type of filter paper for Schleicher and Schuell

GPC:.....Gel Permeation Chromatography

PAH:.....Polynuclear aromatic hydrocarbon (PAH)

HPLC:.....High performance liquid chromatograph

15.0 ATTACHMENTS

Not applicable.

SOUTHWEST LABORATORY OF OKLAHOMA, INC. *and*
AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.

TM

Standard Operating Procedure

Extraction of PCBs by SW846, 3000 Series Methods

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APPROVALS	DATES
Procedure Prepared By	3/7/2001 Date
Program Manager or Section Supervisor	3-8-01 Date
Laboratory Safety Officer	3/12/2001 Date
Laboratory QA/QC Officer	3/9/01 Date

(Effective Date is 5 calendar days after the last signature above - QA/QC Officer)

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EXTRACTION OF PCBs BY SW846, 3000 SERIES METHODS

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1.0 SCOPE AND APPLICATION

1.1 This method is designed to determine the concentrations of various polychlorinated biphenyls (PCBs) as Aroclors™ or individual PCB congeners in extracts from solid and liquid matrices. The method is based on EPA SW-846 methods 8000B and 8082 for analysis and methods 3500B, 3510C, 3520C, 3540C, 3550B, 3580A, 3600C, 3660B, and 3665A for extraction and cleanup. The method can be used to determine analyte concentrations of PCBs in the parts per billion (ppb) range in water and soil matrices. Test codes encompassed by this method include GC601, 602, 610 and GC699.

1.2 A large number of compounds will give response in the electron capture detector (ECD) using this method. Tables 1, 2, and 3 represent the compounds that may be analyzed by this method.

TABLE 1
Aroclor Target Compound List

ANALYTE	CAS #
Aroclor 1016	12674-11-2
Aroclor 1221	11104-28-2
Aroclor 1232	11141-16-5
Aroclor 1242	53469-21-9
Aroclor 1248	12672-29-6
Aroclor 1254	11097-69-1
Aroclor 1260	11096-82-5

TABLE 2
PCB Congener Target Compound List

ANALYTE	CAS #	IUPAC#
2-Chlorobiphenyl	2051-60-7	1
2,3-Dichlorobiphenyl	16605-91-7	5
2,2',5-Trichlorbiphenyl	37680-65-2	18
2,4',5-Trichlorobiphenyl	16606-02-3	31
2,2'3,5'-Tetrachlorobiphenyl	41464-39-5	44
2,2',5,5'-Tetrachlorbiphenyl	35693-99-3	52
2,3',4,4'-Tetrachlorobiphenyl	32598-10-0	66
2,2',3,4,5'-Pentachlorobiphenyl	38380-02-8	87
2,2',4,5,5'-Pentachlorobiphenyl	37680-73-2	101
2,3,3',4',6-Pentachlorobiphenyl	38380-03-9	110
2,2',3,4,4',5'-Hexachlorobiphenyl	35065-28-2	138
2,2',3,4,5,5'-Hexachlorobiphenyl	52712-04-6	141
2,2',3,5,5',6-Hexachlorobiphenyl	52663-63-5	151
2,2'-4,4'-5,5'-Hexachlorobiphenyl	35065-27-1	153
2,2',3,3',4,4',5-Heptachlorobiphenyl	35065-30-6	170
2,2',3,4,4',5,5'-Heptachlorobiphenyl	35065-29-3	180
2,2',3,4,4',5',6-Heptachlorobiphenyl	52663-69-1	183
2,2',3,4',5,5',6-Heptachlorobiphenyl	52663-68-0	187
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	40186-72-9	206

Table 3
Extended List Aroclor Target Compounds

ANALYTE	CAS #
Aroclor 1262	37324-23-5
Aroclor 1268	11100-14-4

2.0 METHOD SUMMARY

2.1 Continuous liquid-liquid or separatory funnel extraction procedures are employed for the extraction of aqueous samples. Sonication or Soxhlet extraction is required for soil/sediment samples. Both neat and diluted organic liquids may be analyzed by direct injection (Method 3580A, Waste Dilution). Sulfuric acid cleanup by method 3665 is mandatory for all samples (water/soil).

2.2 A method blank, laboratory control spike, and a matrix spike/matrix spike duplicate must be prepared for all batches of 20 samples or less.

3.0 HEALTH AND SAFETY

3.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data Sheets (MSDS) is available to all personnel involved in chemical analysis.

3.2 PCBs have been tentatively classified as known or suspected, human or mammalian carcinogens. Primary standards of these toxic compounds <u>must</u> be prepared in a hood. A NIOSH/MESA approved toxic gas respirator <u>must</u> be worn when the analyst handles high concentrations of these toxic compounds.

3.3 Caution should be taken when working with all sample extracts and calibration standards. Samples are of unknown origin and should be considered hazardous. Many of the spiking solutions used, along with chlorinated solvents, are considered carcinogens. Concentrated acids and bases, if not handled properly, can cause severe burns.

3.4 Gloves should be used for the manipulation of all sample extracts and calibration standards and during the preparation of all acid and base solutions. Wear personal protective equipment (PPE) and goggles or face shield when warranted. See the standard operating procedure "Laboratory Safety Plan," SWL-GA-111, for detailed safety procedures.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

4.1 Sample Extraction

4.1.1 Aqueous samples must be extracted within 7 days of collection. Solid samples must be extracted within 14 days of collection.

4.1.2 Extracts must be stored under refrigeration at 4°C (±2°C) and analyzed within 40 days of extraction.

4.2 Sample Storage

4.2.1 The samples must be protected from light and refrigerated at 4°C (±2°C) from the time of receipt until 60 days after delivery to the client of a complete, reconciled sample data package. After 60 days the samples may be disposed of in a manner that complies with all applicable regulations.

4.2.2 The sample must be stored in an atmosphere demonstrated to be free of all potential contaminants.

4.2.3 Samples, sample extracts, and standards must be stored separately.

4.3 Sample Extract Storage

4.3.1 Sample extracts must be protected from light and stored at 4°C (±2°C) until 365 days after delivery to the client of a complete data package.

4.3.2 Sample extracts must be stored in an atmosphere demonstrated to be free of all potential contaminants.

4.3.3 Samples, sample extracts, and standards must be stored separately.

4.4 All containers should be glass jars or bottles with Teflon lids. A minimum of one liter is required for aqueous samples. A minimum of 80g is required for soil samples (125-mL glass jar).

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

5.1 Sources of interference in this method can be grouped into three broad categories: contaminated solvents, reagents or sample processing hardware; contaminated GC carrier gas, parts, column surfaces or detector surfaces; and the presence of co-eluting compounds in the sample matrix to which the ECD will respond. Knowledge of good laboratory practices is assumed, including steps to be followed in routine testing and cleanup of solvents, reagents and sample processing hardware, and instrument maintenance. The discussion that follows focuses on sources of interference associated with the sample matrix and compound classes that represent common sources of interference, particularly phthalate esters, organosulfur compounds, lipids, and waxes. Interferences co-extracted from the samples will vary considerably from sample to sample. While general cleanup techniques are referenced or provided as part of this method, unique samples may require additional cleanup approaches to achieve desired degrees of discrimination and quantitation.

5.2 Interferences by phthalate esters introduced during sample preparation can pose a major problem in pesticide determinations. These materials may be removed prior to analysis using sulfuric acid/permanganate cleanup (3665). Common flexible plastics contain

varying amounts of phthalate esters that are easily extracted or leached from such materials during laboratory operations. Cross-contamination of clean glassware routinely occurs when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled. Interferences from phthalate esters can best be minimized by avoiding contact with all plastic materials and checking all solvents and reagents for phthalate contamination. Exhaustive cleanup of solvents, reagents and glassware may be required to eliminate background phthalate ester contamination.

5.3 The presence of elemental sulfur will result in large peaks that interfere with the detection of later eluting organochlorine pesticides. Method 3660 is suggested for removal of sulfur.

5.4 Differing Aroclor mixtures will co-elute to some degree. Care must be taken to choose non-coeluting peaks for quantitation.

5.5 If oven drying of glassware used for PCB analysis is employed, special care must be taken. PCBs are readily volatilized in the oven and spread to other glassware. Therefore, do not dry glassware from samples containing high concentrations of PCBs with glassware that may be used for trace analyses.

6.0 EQUIPMENT/APPARATUS

6.1 Equipment

Separatory funnel - 2 liter, with Teflon stopcock.
Glass funnel with sodium sulfate.
Pasteur glass pipettes - 1 mL, disposable.
Beakers - 400 mL.
Glass or paper thimble or glass wool – contaminant free.
Boiling chips - Solvent extracted and oven baked, approximately 10/40 mesh (silicon carbide or equivalent).
Balance - Top loading, capable of accurately weighing to the nearest 0.01 g to 100g.
Vials - Glass, 2-mL capacity with Teflon lined screw caps or crimp tops.
Glass scintillation vials - 20 mL, with aluminum lined screw caps.
pH indicator paper - narrow range (pH range of 1-6 and 7-11).
Syringe - 1.0, 2.5, and 10.0 mL.
Graduated cylinder - 1 liter.
Spatula - Stainless steel or Teflon.
Glass funnel
Erlenmeyer flask – 250 mL
Test tube rack
Pyrex glass wool
Volumetric flasks, Class A - 10-mL (optional).

6.2 Apparatus

- Apparatus for grinding dry waste samples.
- Soxhlet extractor – 40 mm ID, with 500 mL round bottom flask.

Drying column – 20 mm ID Pyrex® chromatographic column with Pyrex® glass wool at bottom.

NOTE: Fritted glass discs are difficult to decontaminate after highly contaminated extracts have bee passed through. Columns without frits may be purchased. Use a small pad of Pyrex® glass wool to retain the adsorbent. Pre-wash the glass wool pad with 50 mL of acetone followed by 50 mL of elution solvent prior to packing the column with adsorbent.

- Ultrasonic preparation - A horn type device equipped with a titanium tip, or a device that will give equivalent performance, shall be used.

  Ultrasonic Disrupter - The disrupter must have a minimum power wattage of 300 watts, with pulsing capability. A device designed to reduce the cavitation sound is recommended. Follow the manufacturer's instructions for preparing the disrupter for extraction of samples with low and medium/high concentration.

  Use a 3/4" horn for the low concentration method and a 1/8" tapered microtip attached to a 1/2" horn for the medium/high concentration method.

- Sonabox or equivalent- Recommended with above disrupters for decreasing cavitation sound (Heat Systems - Ultrasonics, Inc., Model 432B or equivalent).

- Apparatus for determining percent dry weight:
  Oven - Drying.
  Desiccator.
  Crucibles - Porcelain or disposable aluminum.

- Vacuum or pressure filtration apparatus.
  Buchner funnel.
  Filter paper - S&S #595 (size 18.5 cm) or equivalent.

- Kuderna-Danish (K-D) apparatus.

  Concentrator tube - 10 mL, graduated (Kontes K-570050-1025 or equivalent). A ground-glass stopper is used to prevent evaporation of extracts.

  Evaporation flask - 500 mL (Kontes K-570001-500 or equivalent). Attach to concentrator tube with springs or clips.

  Snyder column - Three ball macro (Kontes K-503000-0121 or equivalent).

  Springs – ½ inch (Kontes K-662750 or equivalent).

- Solvent vapor recovery system (Kontes K-545000-1006 or K547300-0000, Ace Glass 6614-30, or equivalent).

- Water bath - Heated, with concentric ring cover, capable of temperature control (±5°C). The bath should be used in a hood.

- Continuous liquid-liquid extractor - Equipped with Teflon or glass connecting joints and stopcocks requiring no lubrication (Kontes 584200-0000, 584500-0000, 583250-0000, or equivalent).

- Heating mantle - Rheostat controlled.

- Automated 8-place Glascol shaker or equivalent.
- Turbovap extract concentrator.

7.0 REAGENTS

7.1 Organic-free reagent water. All references to water in this method refer to organic-free reagent water.

7.2 Solvents and reagents: As appropriate for Method 3510C, 3520C, 3540C, 3550B, 3630C, 3640A, or 3660B: n-hexane, methylene chloride, acetone, and isooctane (2,2,4-trimethylpentane). All solvents should be pesticide quality or equivalent, and each lot of solvent should be determined to be phthalate free.

7.3 Sodium sulfate (granular, anhydrous), Na_2SO_4. Purify by heating at 400°C for 4 hours in a shallow tray, or by pre-cleaning the Na_2SO_4 with methylene chloride. If the Na_2SO_4 is cleaned with methylene chloride, a method blank must be analyzed, demonstrating that there is no interference from the Na_2SO_4.

7.4 Sulfuric acid solution (1:1 v:v), H_2SO_4. Slowly add 50mL of H_2SO_4 (sp. Gr. 1.84) to 50mL of organic-free reagent water.

7.5 Sodium hydroxide solution, 10 N: (ACS) Dissolve 40g NaOH in reagent water and dilute to 100mL.

8.0 PROCEDURE

Upon receiving a copy of the LIMS (Laboratory Information Management System) sample log, the samples are retrieved from storage and logged out for extraction by the technician according to the chain-of-custody procedure.

The technician now initiates the automated extraction log sheet (See Attachment 1 for Extraction Log example). The pertinent extraction information is keyed into the Extraction Laboratory Database, which is updated as the extraction procedure progresses.

After the samples are extracted and are ready for departmental transfer, the database-generated extraction log is reviewed and submitted with the sample extracts to the appropriate laboratory for analysis.

8.1 Aqueous Sample Procedure: Aqueous samples may be extracted by the Separatory Funnel method or the Continuous Liquid-Liquid method, depending on the nature and number of the samples. Continuous Liquid-Liquid extraction is not recommended for PCB samples. PCBs may be dechlorinated by continuous liquid-liquid extraction, thus the Separatory Funnel method is preferred. Continuous Liquid-Liquid extraction may be required if: 1) Emulsions prevent extraction by Separatory Funnel, or 2) Sample Holding times will be missed due to over-capacity of samples in-house. If the Continuous Liquid-Liquid method is used, then all spiking solutions must be made in methanol to prevent aldol-condensation products, which interfere with analyses.

8.1.1 Separatory Funnel Extraction (Method 3510C):

This method is applicable to the isolation and concentration of water-insoluble and slightly water-soluble organics in preparation for a variety of chromatographic procedures.

8.1.1.1 Using a 1 liter graduated cylinder, measure 1 liter (nominal) of sample and quantitatively transfer it to the separatory funnel. Rinse the graduated cylinder with methylene chloride and pour into the separatory funnel. If high concentrations are anticipated, a smaller volume may be used and then diluted with organic-free reagent water to 1 liter. The pH of the sample is taken at this time. Pour approximately 10mLs of sample into a scintillation vial, read value, and record in the Extractions Log. Add 1.0mL of the surrogate standard to all samples, spikes, and blanks and mix well. For the sample in each analytical batch selected for spiking, add 1.0mL of the matrix-spiking standard and mix well.

8.1.1.2 After spiking, adjust the pH between 5 and 9, if necessary, using 1:1 sulfuric acid or 10 N sodium hydroxide.

8.1.1.3 Add 60mL of methylene chloride to the separatory funnel.

8.1.1.4 Seal and shake the separatory funnel vigorously for 1-2 minutes with periodic venting to release excess pressure. This process can be performed manually or by an automated 8-place Glascol shaker.

NOTE: Methylene chloride creates excessive pressure very rapidly; therefore, initial venting should be done immediately after the separatory funnel has been sealed and shaken once. The separatory funnel should be vented into a hood to prevent unnecessary exposure of the analyst to solvent vapors.

8.1.1.5 Allow the organic layer to separate from the water phase for a minimum of 10 minutes. If the emulsion interface between layers is more than one-third the size of the solvent layer, filtration of the emulsion through glass wool, centrifugation, or other physical methods may be required. Collect the solvent extract in an Erlenmeyer flask. If the emulsion cannot be broken (recovery of < 80% of the methylene chloride, corrected for the water solubility of methylene chloride), transfer the sample, solvent, and emulsion into the extraction chamber of a continuous extractor and proceed as described in Method 3520C, Continuous Liquid-Liquid Extraction.

8.1.1.6 Repeat the extraction two more times using fresh portions of solvent (Sections 8.1.1.3 – 8.1.1.5). Combine the three solvent extracts.

8.1.1.7 Assemble a Kuderna-Danish (K-D) concentrator (if necessary) by attaching a 10mL concentrator tube to a 500-mL evaporation flask.

8.1.1.8 Dry the extract by passing it through a funnel containing about 100g of anhydrous sodium sulfate. Collect the dried extract in a K-D

concentrator. Rinse the Erlenmeyer flask, which contained the solvent extract, with 20-30mL of methylene chloride and add it to the column to complete the quantitative transfer.

8.1.1.9 Add one or two clean boiling chips to the flask and attach a three-ball Snyder column. Prewet the Snyder column by adding about 1mL of methylene chloride to the top of the column. Place the K-D apparatus on a hot water bath (60-65°C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 45-60 minutes. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume of liquid reaches 10-15mL, allow to cool for approximately 10 minutes.

8.1.1.10 Rinse the Snyder column into the sample extract, and transfer the extract to a scintillation vial. If the water bath is not to be used, exchange the extract using the nitrogen blow-down apparatus as noted in Section 8.1.4.1. If exchange is performed at the water bath, concentrate the extracts to the final volume according to section 8.1.4.2.

8.1.1.11 Proceed to method 3665, sulfuric acid cleanup.

8.1.1.12 If sulfur crystals are a problem, proceed to Method 3660 for sulfur cleanup (see Attachment 2, Sulfur Cleanup).

8.1.1.13 The extract obtained may now be analyzed for analyte content using GC/ECD analysis. Transfer the extract to a vial with a Teflon lined screw-cap or crimp top, and label appropriately.

8.1.2 Continuous Liquid-Liquid Extraction (Method 3520C):

8.1.2.1 Using a 1-liter graduated cylinder, measure out 1 liter (nominal) of sample and quantitatively transfer it to the continuous extractor. Rinse the graduated cylinder with methylene chloride and pour into the continuous extractor. If high concentrations are anticipated, a smaller volume may be used and then diluted with organic-free reagent water to 1 liter. It is recommended that if high analyte concentrations are anticipated, samples should be collected in smaller sample bottles and the whole sample used. The pH of the sample is taken at this time. Pour approximately 10mLs of sample into a scintillation vial, read value, and records in the Extraction Log. Pipette 1.0mL of the surrogate standard spiking solution into each sample into the extractor and mix well. For the sample in each analytical batch selected for spiking, add 1.0mL of the matrix-spiking standard and mix well. Adjust pH between 5-9 if necessary before extraction.

8.1.2.2 Add 250mL of methylene chloride to the liquid-liquid extractor and 200mL to the distilling flask. Add a couple of boiling chips to the flask.

8.1.2.3 Add sufficient water to the extractor to ensure proper operation and extract for 18 hours.

8.1.2.4 Allow to cool, then detach the boiling flask. Dry the extract using the technique referred to in Sections 8.1.1.8.

8.1.2.5 Perform concentration using the Kuderna-Danish (K-D) Technique (Section 8.1.3) or Turbovap (proceed according to manufacturer's instructions). Concentrate, if necessary, by following the appropriate steps in Sections 8.1.4.

8.1.3 K-D Technique:

8.1.3.1 Assemble a Kuderna-Danish (K-D) concentrator by attaching a 10-mL concentrator tube to a 500-mL evaporation flask. Dry the extract by passing it through a glass funnel lined with filter paper and filled with anhydrous sodium sulfate. Collect the dried extract in a K-D concentrator. Rinse the flask that contained the solvent extract with 20-30 mL of methylene chloride and add it to the column to complete the quantitative transfer.

8.1.3.2 Add one or two clean boiling chips to the flask and attach a three-ball Snyder column. Prewet the Snyder column by adding about 1mL of methylene chloride to the top of the column. Place the K-D apparatus on a hot water bath (60-65°C above the boiling point of the solvent) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed. Adjust the vertical position of the apparatus and the water temperature, as required, to complete the concentration in 45-60 minutes. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume of liquid reaches 10-15mL, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes. Remove the Snyder column and rinse the flask and its lower joints into the concentrator tube with 1-2mL of extraction solvent.

8.1.3.3 Solvent exchange may be performed at the nitrogen blowdown or at the hot water bath. If exchange is to be performed at the hot-water bath, it may be initiated when the extract volume is at approximately 10mls. At this point immediately add 10.0mls of the exchange solvent (hexane) at the top of the Snyder column. Concentrate the extract to approximately 10.0mls. Remove the K-D assembly from the water bath and rinse the Snyder column with approximately 1-2mls of Hexane and swirl. Allow the extract to cool and then transfer into a pre-cleaned scintillation vial. The extract is reduced to 2.0mls

and brought back up to 10mls at the blowdown apparatus, and is now ready to be acid washed.

8.1.4 Concentration:

Concentration is accomplished by nitrogen blowdown technique and is used to adjust the extract to the final volume required. Solvent exchange is generally performed using the nitrogen blowdown apparatus.

8.1.4.1 Nitrogen Blowdown Technique for Solvent Exchange

8.1.4.1.1 Place the scintillation vial under the nitrogen stream and evaporate the solvent volume to a final volume of 2.0mL using a gentle stream of clean, dry nitrogen (filtered through a column of activated carbon).

CAUTION: Plastic tubing must not be used between the carbon trap and the sample, since it may introduce interferences.

8.1.4.1.2 Add 10mL of hexane to the extract and reduce the volume to 2mL.

8.1.4.1.3 Transfer the extract into a 12-mL vial, rinsing the vial with hexane to ensure quantitative transfer, and bring to the desired final volume of 10.0mL.

8.1.4.1.4 Proceed to method 3665, sulfuric acid cleanup.

8.1.4.1.5 If sulfur crystals are a problem, proceed to Method 3660 for sulfur cleanup.

8.1.4.1.6 The extract obtained may now be analyzed for analyte content using GC/ECD analysis. Transfer the extract to a vial with a Teflon lined screw-cap or crimp top, and label appropriately.

8.1.4.2 Nitrogen Blowdown Technique for Concentration if exchange is performed at the hot water bath.

8.1.4.2.1 Place the scintillation vial under the nitrogen stream and evaporate the solvent volume to a final volume of 10.0mL using a gentle stream of clean, dry nitrogen (filtered through a column of activated carbon).

CAUTION: Plastic tubing must not be used between the carbon trap and the sample, since it may introduce interferences.

8.1.4.2.2 Proceed to method 3665, sulfuric acid cleanup.

8.1.5 Sulfuric Acid Cleanup (Method 3665)

8.1.5.1 Using a syringe, volumetric or disposable pipette, transfer the 10ml Hexane extract to a 20ml scintillation vial and, in a fume hood, carefully add 2mls of concentrated sulfuric acid.

CAUTION: Make sure that there is neither exothermic reaction nor evolution of gas prior to proceeding

8.1.5.2 Cap the vial tightly and vortex and or shake using a wrist-action shaker for 3 minutes. (A vortex must be visible in the vial)

NOTE: A wrist-action shaker is mandatory for this procedure.

CAUTION: Stop the vortexing or wrist-action shaking immediately if the vial leaks.

NOTE: Avoid skin contact: sulfuric acid burns!

8.1.5.3 Allow the phases to separate for at least 1 minute. Examine the top (hexane) layer; it should not be highly colored nor should it have a visible emulsion or cloudiness.

8.1.5.4 If a clean phase separation is achieved, proceed to transfer and submitting the extract.

8.1.5.5 If the hexane layer is colored or the emulsions persists for several minutes, remove the sulfuric acid layer (bottom layer) from the vial and dispose of it properly. Add another 2ml portion of the clean concentrated sulfuric acid, and perform another acid cleanup (wrist-action shaker).

NOTE: Do not remove any hexane from the vial at this stage of the procedure.

8.1.5.6 If the hexane layer is still colored, the analyst may proceed either to another acid cleanup or to the Permanganate cleanup procedure.

8.1.5.7 If the hexane layer is clean (clear) transfer the extract to a clean 12ml vial and submit to the GC Department for analysis along with a completed computerized extraction sheet.

8.1.5.8 The extract obtained may now be analyzed for analyte content using GC/ECD analysis. Transfer the extract to a vial with a Teflon lined screw cap or crimp top, and label appropriately.

8.1.6 Permanganate Cleanup:

8.1.6.1 The permanganate cleanup should be employed when the sulfuric acid cleanup has not removed all of the color from an extract.

8.1.6.2 Add 5ml of the 5% aqueous potassium permanganate solution to the hexane fraction.

CAUTION: Make sure that there is no exothermic reaction nor evolution of gas prior to proceeding.

8.1.6.3 Cap the vial tightly and shake for 3 minutes, using a wrist-action shaker.

CAUTION: Stop the shaking immediately if the vial leaks. Avoid skin contact, potassium permanganate burns.

8.1.6.4 Allow the phases to separate for at least 1 minute. Examine the top (hexane) layer, it should not be highly colored nor should it have a visible emulsion or cloudiness.

8.1.6.5 If a clear phase separation is achieved proceed to removing the hexane layer and submitting the extract.

8.1.6.6 If the hexane layer is colored or the emulsion persists for several minutes, remove the permanganate solution from the vial via a glass pipette and dispose of the clean aqueous permanganate solution.

NOTE: Do not remove any hexane at this stage of the procedure.

8.1.6.7 Shake the sample and allow the phases to separate.

8.1.6.8 Transfer the hexane layer to a clean 12ml vial and submit for analysis.

8.2 Solid Sample Procedure: Solid samples may be extracted based on method 3550B, Ultrasonic Extraction, or method 3540C, Soxhlet extraction. The ultrasonic procedure is divided into two sections, based on the expected concentration of organics in the sample. The low concentration method (individual organic components of ≤ 20 mg/kg) uses a larger sample size and a more rigorous extraction procedure (lower concentrations are more difficult to extract). The medium/high concentration method (individual organic components of > 20 mg/kg) is much simpler and therefore faster. It is highly recommended that the extracts be cleaned up prior to analysis.

8.2.1 Sample Handling:

8.2.1.1 Sediment/soil samples — Decant and discard any water layer on sediment sample. Mix sample thoroughly, especially composited samples. Discard any foreign objects such as sticks, leaves, and rocks.

8.2.1.2 Determine the dry weight of the sample remaining after decanting. Measurement of soil pH is required.

8.2.1.3 Waste samples — Samples consisting of multiphases must be prepared by the phase separation method in Chapter Two of SW-846 before extraction. This procedure is for solids only.

8.2.1.4 Dry waste samples amenable to grinding; grind or otherwise subdivide the waste.

8.2.1.5 Gummy, fibrous or oily materials not amenable to grinding should be cut, shredded, or otherwise broken up to allow mixing, and maximum exposure of the sample surfaces for extraction. The professional judgment of the analyst is required for handling of these difficult matrices. The addition of anhydrous sodium sulfate to the sample (1:1) may make the mixture amenable to grinding.

8.2.1.6 Determination of percent moisture: weigh out a portion of sample for this determination at the same time as the portion used for analytical determination.

WARNING: The drying oven should be contained in a hood or vented. Significant laboratory contamination may result from drying a heavily contaminated hazardous waste sample. However, samples known or suspected to contain significant concentrations of toxic, flammable, or explosive constituents should not be over-dried because of concerns for personal safety. Laboratory discretion is advised. It may be prudent to delay oven-drying of the weighed-out portion until other analytical results are available.

8.2.2 Low concentrations of organics (≤ 20 mg/kg; Method 3550B):

8.2.2.1 The following step should be performed rapidly to avoid loss of the more volatile extractables. Weigh approximately 30g of sample into a 400-mL beaker. Record the weight to the nearest 0.1g (DO NOT TARGET WEIGHTS). Nonporous or wet samples (gummy or clay type) that do not have a free flowing sandy texture must be mixed with approximately 60g of anhydrous sodium sulfate, using a spatula. After addition of sodium sulfate, the sample should be free flowing. Add 1.0mL of pesticide surrogate standards to all samples, spikes, standards, and blanks. For the sample in each analytical batch selected for spiking, add 1.0mL of the pesticide matrix-spiking standard. Immediately add 100mL of 1:1 methylene chloride/acetone.

8.2.2.2 Place the bottom surface of the tip of the disrupter horn about ½ inch below the surface of the solvent, but above the sediment layer. Be sure that the horn is properly tuned according the manufacturer's instructions.

8.2.2.3 Extract ultrasonically for 3 minutes, with output control knob set at 10 (full power) and with mode switch on Pulse (pulsing energy rather than continuous energy) and percent-duty cycle knob set at 50% (energy on 50% of time and off 50% of time). <u>Do not use microtip probe</u>.

8.2.2.4 Decant the extract and filter it through S&S 595 size 18.5 cm filter paper (or equivalent) in a glass funnel filled with sodium sulfate, if needed.

8.2.2.5 Repeat the extraction two or more times with two additional 100mL portions of the solvent mixture. Decant off the solvent after each ultrasonic extraction. On the final ultrasonic extraction, pour the entire sample into the funnel and rinse with extraction solvent. Apply a vacuum to the filtration flask, and collect the solvent extract. Continue filtration until all visible solvent is removed from the funnel, but do not attempt to completely dry the sample, as the continued application of a vacuum may result in the loss of some analytes. Alternatively, if centrifugation is used, transfer the entire sample to the centrifuge bottle. Centrifuge at low speed, and then decant the solvent from the bottle.

8.2.2.6 Assemble a Kuderna-Danish (K-D) concentrator by attaching a 10-mL concentrator tube to a 500-mL evaporator flask. Transfer filtered extract to a 500-mL evaporator flask and proceed to the next section.

8.2.2.7 Add one or two clean boiling chips to the flask and attach a three-ball Snyder column. Prewet the Snyder column by adding about 1mL of 1:1 methylene chloride/acetone to the top of the column. Place the K-D apparatus on a hot water bath (60-75°C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 45-60 minutes. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume of liquid reaches 10-15 mL, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes.

8.2.2.8 Rinse and remove the Snyder column, and transfer the extract to a scintillation vial. Exchange (if necessary) and concentrate the extract, using the appropriate nitrogen blowdown technique as outlined in Sections 8.1.4.1 through 8.1.4.2, or the solvent may be exchanged at the hot water bath using the technique as outlined in Section 8.1.3.3.

8.2.2.9 Sulfuric acid cleanup is now employed for all samples, QC and method blanks.

8.2.2.10 If sulfur crystals are a problem, proceed to Method 3660 for cleanup.

8.2.2.11 The extract obtained may now be analyzed for analyte content using GC/ECD analysis. Transfer the extract to a vial with Teflon lined screw caps or crimp the top, and label appropriately.

8.2.3 Medium/high concentration PCB method (individual organic components of > 20 mg/kg):

8.2.3.1 Transfer approximately 1g (record weight to the nearest 0.1g) of sample to a 20mL vial. Wipe the mouth of the vial with a tissue to remove any sample material. Record the exact weight of sample

taken. Cap the vial before proceeding with the next sample to avoid any cross contamination.

8.2.3.2 Add 2g of anhydrous sodium sulfate to sample in the 20-mL vial and mix well.

8.2.3.3 Surrogate standards are added to all samples, spikes, and QC. Add 1.0mL of pesticide surrogate spiking solution to sample mixture. For the sample in each analytical batch selected for spiking, add 1.0mL of the pesticide matrix-spiking standard.

8.2.3.4 Immediately add whatever volume of solvent is necessary to bring the final volume to 10.0mL considering the added volume of surrogates and matrix spikes. Disrupt the sample with the 1/8 inch tapered microtip ultrasonic probe for 3 minutes at output control setting 5 and with mode switch on pulse and percent duty cycle at 50%. The extraction solvent for PCBs is hexane.

8.2.3.5 Loosely pack disposable Pasteur pipettes with 2 to 3cm Pyrex glass wool plugs. Filter the extract through the glass wool and collect the extract in a scintillation vial.

8.2.3.6 Sulfuric acid cleanup is now employed for all samples, QC and method blanks.

8.2.3.7 The extract obtained may now be analyzed for analyte content using GC/ECD analysis. Transfer the extract to a vial with Teflon lined screw-caps or crimp the top, and label appropriately.

8.2.4 Soxhlet Extraction (Method 3540C):

8.2.4.1 Immediately after weighing the sample for extraction, weigh 5-10g of the sample into a disposable aluminum weighing boat. Determine the % moisture of the sample by drying at 105°C-115°C (±2°C) for a predetermined amount of time or over night. Allow to cool before weighing. See Section 9.0, "Calculations" for calculation of percent moisture.

8.2.4.2 Blend 30g of the solid sample with 60g of anhydrous sodium sulfate and place in an extraction thimble. The extraction thimble must drain freely for the duration of the extraction period. A glass wool plug above and below the sample in the Soxhlet extractor is an acceptable alternative for the thimble. Add 1.0mL of the surrogate standard spiking solution onto the sample. For the sample in each analytical batch selected for spiking, add 1.0mL of the matrix-spiking standard.

8.2.4.3 Place approximately 400-450mL of the extraction solvent (methylene chloride) into a 500mL round bottom flask containing one or two clean boiling chips. Attach the flask to the extractor and extract the sample for 18 hours at 4-6 cycles/hr.

8.2.4.4 Allow the extract to cool after the extraction is complete.

8.2.4.5 Perform concentration (if necessary) using the Kuderna-Danish (K-D) Technique (Section 8.1.3) or Turbovap (proceed according to manufacturer's instructions). See Sections 8.1.4 for exchange (if necessary) and/or extract concentration.

8.2.4.6 Sulfuric acid cleanup is now employed for all samples, QC and method blanks (see 8.1.5).

8.2.4.7 If sulfur crystals are a problem, proceed to Method 3660 for cleanup.

8.2.4.8 The extract obtained may now be analyzed for analyte content using GC/ECD analysis. Transfer the extract to a vial with Teflon lined screw-caps or crimp the top, and label appropriately.

8.2.5 Waste Dilution

8.2.5.1 This method describes a solvent dilution of a non-aqueous waste sample prior to cleanup and/or analysis. It is designed for wastes that may contain organic chemicals at a concentration greater than 20,000 mg/Kg and that are soluble in the dilution solvent.

8.2.5.2 Samples consisting of multiphases must be prepared by the phase separation method before extraction.

8.2.5.3 The sample dilution may be performed in a 10-mL volumetric flask. If disposable glassware is preferred, the 20-mL scintillation vial may be calibrated for use. Pipette 10.0mL of extraction solvent into the scintillation vial and mark the bottom of the meniscus. Discard this solvent.

8.2.5.4 Transfer approximately 1 g of each phase of the sample to separate 20mL vials or 10-mL volumetric flasks (record weight to the nearest 0.1 g). Wipe the mouth of the vial with a tissue to remove any sample material. Cap the vial before proceeding with the next sample to avoid any cross-contamination.

8.2.5.5 Add 2.0mL surrogate spiking solution to all samples and blanks. For the sample in each analytical batch selected for spiking, add 2.0mL of the matrix-spiking standard.

8.2.5.6 Immediately dilute to 10mL with hexane.

8.2.5.7 Add 2.0g of anhydrous sodium sulfate to the sample.

8.2.5.8 Cap and shake the sample for 2 min.

8.2.5.9 Loosely pack disposable Pasteur pipettes with 2-3 cm glass wool plugs. Filter the extract through the glass wool and collect 5 mL of the extract in a tube or vial.

8.2.5.10 The extract is then ready for cleanup.

8.2.5.11 Sulfuric acid cleanup is now employed for all samples, QC and method blanks. The 10mL extract is then cleaned by shaking out with concentrated sulfuric acid solution for several minutes (wrist action shaker recommended). Allow the phases to separate and examine the hexane layer (top layer). If the hexane layer is colored or has a visible emulsion or cloudiness, draw off the hexane layer and repeat the procedure until no color or cloudiness remains in the extract. The hexane layer is then drawn off. The clean extract is then submitted to the Gas Chromatography (GC) Laboratory.

8.2.5.12 If sulfur crystals are a problem, proceed to Method 3660 for cleanup.

8.2.5.13 The extract obtained may now be analyzed for analyte content using GC/ECD analysis. Transfer the extract to a vial with Teflon lined screw caps or crimp the top, and label appropriately.

8.2.5.14 Any reagent blanks and matrix spike samples must be subjected to exactly the same analytical procedures as those used on actual samples.

8.2.6 Sulfuric Acid Cleanup (Method 3665)

8.2.6.1 Using a syringe, volumetric or disposable pipette, transfer the 10ml Hexane extract to a 20ml scintillation vial and, in a fume hood, carefully add 2mls of concentrated sulfuric acid.

CAUTION: Make sure that there is neither exothermic reaction nor evolution of gas prior to proceeding.

8.2.6.2 Cap the vial tightly and vortex and or shake using a wrist-action shaker for 3 minutes. (A vortex must be visible in the vial)

NOTE: A wrist-action shaker is mandatory for this procedure.

CAUTION: Stop the vortexing or wrist-action shaking immediately if the vial leaks.

NOTE: Avoid skin contact: sulfuric acid burns!

8.2.6.3 Allow the phases to separate for at least 1 minute. Examine the top (hexane) layer; it should not be highly colored nor should it have a visible emulsion or cloudiness.

8.2.6.4 If a clean phase separation is achieved, proceed to transfer and submitting the extract.

8.2.6.5 If the hexane layer is colored or the emulsions persists for several minutes, remove the sulfuric acid layer (bottom layer) from the vial and dispose of it properly. Add another 2ml portion of the clean

concentrated sulfuric acid, and perform another acid cleanup (wrist-action shaker).

NOTE: Do not remove any hexane from the vial at this stage of the procedure.

8.2.6.6 If the hexane layer is still colored, the analyst may proceed either to another acid cleanup or to the Permanganate cleanup procedure.

8.2.6.7 If the hexane layer is clean (clear) transfer the extract to a clean 12ml vial and submit to the GC Department for analysis along with a completed computerized extraction sheet.

8.2.6.8 The extract obtained may now be analyzed for analyte content using GC/ECD analysis. Transfer the extract to a vial with a Teflon lined screw cap or crimp top, and label appropriately.

9.0 CALCULATIONS

Not applicable.

10.0 QUALITY ASSURANCE/QUALITY CONTROL

10.1 Every 20 samples or sample delivery group (SDG) is considered a batch. Two method blanks, laboratory control spikes, and a matrix spike (MS) and matrix spike duplicate (MSD) are extracted with each SDG of up to 20 samples. In most cases, a client will specify a sample for MS/MSD. If MS/MSD sample is not specified, the laboratory chooses one at their discretion (Field Blanks excluded). Also, if two or more clients send samples not totaling 20 in number, one set of method blanks, LCS and MS/MSD are applied to both clients' samples.

10.2 Any reagent blanks or matrix spike samples should be subjected to exactly the same analytical procedures as those used on actual samples.

10.3 Refer to Chapter One of SW-846 for specific quality control procedures and Method 3500 for extraction and sample preparation procedures.

11.0 WASTE DISPOSAL / POLLUTION PREVENTION

See SOP SWL-GA-114, "*Hazardous Waste Management Plan.*"

12.0 METHOD PERFORMANCE

See SOP SWL-OP-415, "*Analysis of PCBs by GC, SW-846, Method 8082.*"

13.0 REFERENCES

13.1 U.S. EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule," October 26, 1984.

13.2 U.S. EPA SW-846, "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", Revision 4, December 1996

14.0 DEFINITIONS

Not applicable.

15.0 ATTACHMENTS

Attachment 1: Extraction Log Sheet

Attachment 2: Method 3660B — Sulfur Cleanup

ATTACHMENT 1
Extraction Log Sheet

(3520C)
SEP-F
(3510C) XXXXX
SHAKER
SONC.
(3550B)
SOXHLET

BATCH: 000831WC

EXTRACT DATE 2000-08-31 12:38

001748

SAMPLE ID (EPISODE #)	CLIENT CASE	DATE RECEIVED CLIENT SAMPLE ID	MATRIX/LEVEL	SAMPLE pH	% MOISTURE %	DRY WEIGHT g	AMOUNT EXTRACTED g/ml	EXTRACTED VOLUME ml	GPC VOLUME ml	GPC ELUTE VOL ml	FLORISIL VOL CONC ml	FLORISIL FIN VOL CONC ml	VOL SV (P) ml	FIN PROCESS VOLUME ml	SCREEN Y/N Y/N	MS SV (P) µL	SURR SV (P) µL	BASE pH CONFIRMATION	ACID pH CONFIRMATION	Bottle
BL0831WC			W/L				1000	300					10.0	10.0	N		1000			
BB0831WC			W/L				1000	300					10.0	10.0	N		1000			
LC0831WC			W/L				1000	300					10.0	10.0	N	1000	1000			
LD0831WC			W/L				1000	300					10.0	10.0	N	1000	1000			
44130.04 GC601	BECJACOB	08/29/2000 10:00 SA22	W/L	7.11			1000	300					10.0	10.0	N		1000			A
44130.05MS GC601	BECJACOB	08/29/2000 10:00 SA22	W/L	7.11			1000	300					10.0	10.0	N	1000	1000			AB
44130.06MSD GC601	BECJACOB	08/29/2000 10:00 SA22	W/L	7.10			1000	300					10.0	10.0	N	1000	1000			AB
44130.17 GC601	BECJACOB	08/29/2000 10:00 SA36	W/L	8.01			1000	300					10.0	10.0	N		1000			A
44130.22 GC601	BECJACOB	08/29/2000 10:00 SA92	W/L	6.92			1000	300					10.0	10.0	N		1000			A
44130.27 GC601	BECJACOB	08/29/2000 10:00 SA175	W/L	7.38			1000	300					10.0	10.0	N		1000			A
44130.32 GC601	BECJACOB	08/29/2000 10:00 SA8	W/L	7.10			1000	300					10.0	10.0	N		1000			A
44130.37 GC601	BECJACOB	08/29/2000 10:00 SA64	W/L	7.06			1000	300					10.0	10.0	N		1000			B
44151.01 GC601	B&MC	08/30/2000 09:50 LTANK1	W/L	7.48			1000	300					10.0	10.0	N		1000			A

INITIALS

WEIGHING:	SA	EXTRACTION:	JM	FLORISIL:	MeCl2 Hexane
CLEANUP:		KD:	CD		
BLOWDOWN:	JM	TV:			
SURROG. SPIKER:	JM	VERIFIER:	GS		
MATRIX SPIKER:	JM	VERIFIER:	GS		
SUBMITTED BY:	JM	REVIEWED BY:			

ID NUMBERS

FLORISIL CARTRIDGE #:

GPC COLUMN #:

SURROGATES ID CONC #: 5-218-5

MATRIX SPIKE SOLID/CONC #(s): 5-219-5

SOP - METHOD # : SWL-OP- 415 Rev. 3.0

COMMENTS

Acid washed

Pg 2 BK1 Pg8

SEP-F (3510C)
SHAKER
SONC. (3550B) XXXXX
SOXHLET

BATCH: 0006

EXTRACT DATE: 2000-08-30 15:42

001743
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SAMPLE ID (EPISODE #)	CLIENT CASE	DATE RECEIVED CLIENT SAMPLE ID	MATRIX/LEVEL	SAMPLE pH	% MOISTURE %	DRY WEIGHT g	AMOUNT EXTRACTED g*(ml)	EXTRACTED VOLUME ml	GPC VOLUME ml	GPC ELUTE VOL ml	FLORISIL VOL CONC ml	FLORISIL FIN VOL CONC ml	VOL SV (P) ml	FIN PROCESS VOLUME ml	SCREEN Y/N Y/N	MS SV (P) µL	SURR SV (P) µL	BASE pH CONFIRMATION	ACID pH CONFIRMATION	
BL0830SH			S/L				30.00	350					10.0	10.0	N		1000			Bottle
BB0830SH			S/L				30.00	350					10.0	10.0	N		1000			
LC0830SH			S/L				30.00	350					10.0	10.0	N	1000	1000			
LD0830SH			S/L				30.00	350					10.0	10.0	N	1000	1000			
44140.01 GC601	ESCVA	08/30/2000 12:35 D 7	S/L	6.59	16.62	25.1	30.15	350					10.0	10.0	N		1000			A
44140.01MS GC601	ESCVA	08/30/2000 12:35 D 7	S/L	6.59	16.62	25.1	30.08	350					10.0	10.0	N	1000	1000			A
44140.01MSD GC601	ESCVA	08/30/2000 12:35 D 7	S/L	6.59	16.62	25.0	30.01	350					10.0	10.0	N	1000	1000			A

INITIALS

WEIGHING:	CD	EXTRACTION:	CD	FLORISIL:	CH_2Cl_2/acetone Hexane
CLEANUP:	CD	KD:	CD		
BLOWDOWN:	CD	TV:			
SURROG. SPIKER:	CD	VERIFIER:	JM		
MATRIX SPIKER:	CD	VERIFIER:	JM		
SUBMITTED BY:	CD	REVIEWED BY:	8/31/00		

ID NUMBERS

FLORISIL CARTRIDGE #:
GPC COLUMN #:
SURROGATES ID CONC #: 5-218-5
MATRIX SPIKE SOLID/CONC #(s): 5-219-5
SOP - METHOD # : SWL-OP- 416 Rev. 3.0

COMMENTS

Acid Washed

Pg 29 Bk 1 Reagent

ATTACHMENT 2
Method 3660B — Sulfur Cleanup

SULFUR CLEANUP

1.0 SCOPE AND APPLICATION

1.1 Elemental sulfur is encountered in many sediment samples (generally specific to different areas in the country), marine algae, and some industrial wastes. The solubility of sulfur in various solvents is very similar to the organochlorine and organophosphorus pesticides. Therefore, the sulfur interference follows along with the pesticides through the normal extraction and cleanup techniques. In general, sulfur will usually elute entirely in Fraction 1 of the Florisil cleanup (Method 3620).

1.2 Sulfur will be quite evident in gas chromatograms obtained from electron capture detectors, flame photometric detectors operated in the sulfur or phosphorous mode, and Coulson electrolytic conductivity detectors in the sulfur mode. If the gas chromatograph is operated at the normal conditions for pesticide analysis, the sulfur interference can completely mask the region from the solvent peak through Aldrin.

1.3 Two techniques for the elimination of sulfur are detailed within this method: (1) the use of copper powder; and (2) the use of tetrabutylammonium sulfite. Tetrabutylammonium sulfite causes the least amount of degradation of a broad range of pesticides and organic compounds, while copper may degrade organophosphorus and some organochlorine pesticides.

1.4 This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

2.1 The sample to undergo cleanup is mixed with either copper or tetrabutylammonium (TBA) sulfite. The mixture is shaken and the extract is removed from the sulfur cleanup reagent.

3.0 INTERFERENCES

The copper technique requires that the copper powder be very reactive, as evidenced by a bright shiny appearance (see Sec. 5.5 for the preparation of this reagent). However, care must be taken to remove all traces of the acid used to prepare the copper, in order to avoid degradation of some analytes.

4.0 APPARATUS AND MATERIALS

4.1 Mechanical shaker or mixer - Vortex Genie or equivalent.

4.2 Pipets, disposable - Pasteur type.

4.3 Centrifuge tubes, calibrated - 12 mL.

4.4 Glass bottles or vials - 10 mL and 50 mL, with polytetrafluoroethylene (PTFE)-lined screw caps or crimp tops.

5.0 REAGENTS

5.1 Reagent grade 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. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Organic-free reagent water - All references to water in this method refer to organic-free reagent water, as defined in Chapter One.

5.3 Nitric acid, HNO_3, dilute.

5.4 Solvents

5.4.1 Acetone, CH_3COCH_3 - Pesticide quality or equivalent.

5.4.2 Hexane, C_6H_{14} - Pesticide quality or equivalent.

5.4.3 2-Propanol, $CH_3CH(OH)CH_3$ - Pesticide quality or equivalent.

5.5 Copper powder - Remove oxides by treating with dilute nitric acid, rinse with organic-free reagent water to remove all traces of acid, rinse with acetone and dry under a stream of nitrogen. (Copper, fine granular Mallinckrodt 4649 or equivalent).

5.6 Tetrabutylammonium (TBA) sulfite reagent

5.6.1 Tetrabutylammonium hydrogen sulfate, $[CH_3(CH_2)_3]_4NHSO_4$.

5.6.2 Sodium sulfite, Na_2SO_3.

5.6.3 Prepare reagent by dissolving 3.39 g tetrabutylammonium hydrogen sulfate in 100 mL organic-free reagent water. To remove impurities, extract this solution three times with 20 mL portions of hexane. Discard the hexane extracts, and add 25 g sodium sulfite to the water solution. Store the resulting solution, which is saturated with sodium sulfite, in an amber bottle with a PTFE-lined screw cap. This solution can be stored at room temperature for at least one month.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 See the introductory material to this chapter, Organic Analytes, Sec. 4.1.

7.0 PROCEDURE

7.1 Removal of sulfur using copper.

7.1.1 Concentrate the sample to exactly 1.0 mL or other known volume. Perform concentration using the techniques described in the appropriate 3500 series method.

CAUTION: When the volume of solvent is reduced below 1 mL, semivolatile analytes may be lost.

7.1.2 If the sulfur concentration is such that crystallization occurs, centrifuge to settle the crystals, and carefully draw off the sample extract with a disposable pipet leaving the excess sulfur in the concentration vessel. Transfer 1.0 mL of the extract to a calibrated centrifuge tube.

7.1.3 Add approximately 2 g of cleaned copper powder to the centrifuge tube. (The copper will fill the tube to approximately the 0.5 mL mark). Vigorously mix the extract and the copper powder for at least 1 min on the mechanical shaker. Allow the phases to separate.

7.1.4 Separate the extract from the copper by drawing off the extract with a disposable pipet and transfer to a clean vial. The volume remaining still represents 1.0 mL of extract.

NOTE: This separation is necessary to prevent further degradation of the pesticides.

7.2 Removal of sulfur using TBA sulfite

7.2.1 Concentrate the sample extract to exactly 1.0 mL or other known volume. Perform concentration using the techniques described in the appropriate 3500 series method.

CAUTION: When the volume of solvent is reduced below 1 mL, semivolatile analytes may be lost.

7.2.2 Transfer 1.0 mL of the extract to a 50 mL clear glass bottle or vial with a PTFE-lined screw-cap. Rinse the concentrator tube with 1 mL of hexane, adding the rinsings to the 50 mL bottle.

7.2.3 Add 1.0 mL TBA sulfite reagent and 2 mL 2-propanol, cap the bottle, and shake for at least 1 min. If the sample is colorless or if the initial color is unchanged, and if clear crystals (precipitated sodium sulfite) are observed, sufficient sodium sulfite is present. If the precipitated sodium sulfite disappears, add more crystalline sodium sulfite in approximately 0.100 g portions until a solid residue remains after repeated shaking.

7.2.4 Add 5 mL organic free reagent water and shake for at least 1 min. Allow the sample to stand for 5-10 min. Transfer the hexane layer (top) to a concentrator tube and concentrate the extract to approximately 1.0 mL using the techniques described in the appropriate 3500 series method. Record the actual volume of the final extract.

7.3 Analyze the cleaned up extracts by gas chromatography (see the determinative methods, Sec. 4.3 of this chapter).

8.0 QUALITY CONTROL

8.1 Refer to Chapter One for specific quality control procedures and Method 3600 for cleanup procedures.

8.2 All reagents should be checked prior to use to verify that interferences do not exist.

9.0 METHOD PERFORMANCE

9.1 Table 1 indicates the effect of using copper to remove sulfur on the recovery of certain pesticides.

10.0 REFERENCES

1. Goerlitz, D.F. and L.M. Law, Bulletin for Environmental Contamination and Toxicology, 6, 9 (1971).

2. U.S. EPA Contract Laboratory Program, Statement of Work for Organic Analysis, Revision, July 1985.

SOUTHWEST LABORATORY OF OKLAHOMA, INC. *and*
AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.

TM

Standard Operating Procedure

Extraction of Pesticides by SW846, 3000 Series Methods

Document No.: SWL-OE-408

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APPROVALS	DATES
Procedure Prepared By	3/7/2001 Date
Program Manager or Section Supervisor	3-8-01 Date
Laboratory Safety Officer	3/12/01 Date
Laboratory QA/QC Officer	3/9/01 Date

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EXTRACTION OF PESTICIDES BY SW846, 3000 SERIES METHODS

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1.0 SCOPE AND APPLICATION

This method is designed to determine the concentrations of various organochlorine pesticides by extraction of the compounds from solid and liquid matrices. The method is based on EPA SW-846 and methods 3500B, 3510C, 3520C, 3540C, 3550B, 3580A, 3600C, 3610B, 3620B, 3640A, and 3660B for extraction and cleanup. The methods can be used to determine analyte concentrations of pesticides in the ppt range in water. Test codes encompassed by this method include but are not limited to GC801 and GC899.

A large number of compounds will give response in the electron capture detector (ECD) using this method. Tables 1 and 2 represent the compounds that may be analyzed by this method.

Table 1
Organochlorine Pesticide Target Compounds List

ANALYTE	CAS #
Aldrin	309-00-2
Alpha-BHC	319-84-6
Beta-BHC	319-85-7
Delta-BHC	319-86-8
Gamma-BHC	58-89-9
alpha-Chlordane	5103-71-9
gamma-Chlordane	5103-74-2
4,4'-DDD	72-54-8
4,4'-DDE	72-55-9
4,4'-DDT	50-29-3
Dieldrin	60-57-1
Endosulfan I	959-98-8
Endosulfan II	33212-65-9
Endosulfan Sulfate	1031-07-8
Endrin	72-20-8
Endrin Aldehyde	7421-93-4
Endrin Ketone	53494-70-5
Heptachlor	76-44-8
Heptachlor Epoxide	1024-57-3
4,4'-Methoxychlor	72-43-5
Toxaphene	8001-35-2

Table 2
Organochlorine Pesticide Target Compounds List (Extended List)

ANALYTE	CAS #
Aldrin	309-00-2
Alpha-BHC	319-84-6
Beta-BHC	319-85-7
Delta-BHC	319-86-8
Gamma-BHC	58-89-9
alpha-Chlordane	5103-71-9
gamma-Chlordane	5103-74-2
4,4'-DDD	72-54-8
4,4'-DDE	72-55-9
4,4'-DDT	50-29-3
Dieldrin	60-57-1
Endosulfan I	959-98-8
Endosulfan II	33212-65-9
Endosulfan Sulfate	1031-07-8
Endrin	72-20-8
Endrin Aldehyde	7421-93-4
Endrin Ketone	53494-70-5
Heptachlor	76-44-8
Heptachlor Epoxide	1024-57-3
4,4'-Methoxychlor	72-43-5
Toxaphene	8001-35-2
Chlorobenzilate	510-15-6
DBCP	96-12-8
Diallate	2303-16-4
Hexachlorobenzene	118-74-1
Hexachlorocyclopentadiene	77-47-4
Isodrin	465-73-6

2.0 METHOD SUMMARY

Continuous liquid-liquid or separatory funnel extraction procedures are employed for the extraction of aqueous samples. Sonication or Soxhlet extraction is required for soil/sediment samples. Gel Permeation (GPC) and florisil or alumina columns may be used for cleanup of extracts. Both neat and diluted organic liquids may be analyzed by direct injection (Method 3580A, Waste Dilution).

Continuous liquid-liquid extraction is not a recommended sample preparation method for Method 8081A analyses. Organochlorine pesticides many dechlorinate. Therefore, method 3510C is recommended for 8081A analyses. Method 3520C may be used in certain instances if pre-approved by management.

3.0 HEALTH AND SAFETY

3.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data Sheets (MSDS) is available to all personnel involved in chemical analysis.

3.2 The following parameters covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: 4,4'-DDT, 4.4'-DDD, the BHCs, and the PCBs. Primary standards of these toxic compounds must be prepared in a hood. A NIOSH/MESA approved toxic gas respirator must be worn when the analyst handles high concentrations of these toxic compounds.

3.3 Caution should be taken when working with all sample extracts and calibration standards. Samples are of unknown origin and should be considered hazardous. Many of the spiking solutions used, along with chlorinated solvents, are considered carcinogens. Concentrated acids and bases, if not handled properly, can cause severe burns.

3.4 Gloves should be used for the manipulation of all sample extracts and calibration standards and during the preparation of all acid and base solutions. Wear personal protective equipment (PPE) and goggles or face shield when warranted. See the standard operating procedure for "Laboratory Safety Plan", SWL-GA-111 for detailed safety procedures.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

4.1 Sample Extraction

4.1.1 Aqueous samples must be extracted within 7 days of collection. Solid samples must be extracted within 14 days of collection.

4.1.2 Extracts must be stored under refrigeration at 4°C (±2°C) and analyzed within 40 days of extraction.

4.2 Sample Storage

4.2.1 The samples must be protected from light and refrigerated at 4°C (±2°C) from the time of receipt until 60 days after delivery to the client of a complete, reconciled sample data package. After 60 days the samples may be disposed of in a manner that complies with all applicable regulations.

4.2.2 The sample must be stored in an atmosphere demonstrated to be free of all potential contaminants.

4.2.3 Samples, sample extracts, and standards must be stored separately.

4.3 Sample Extract Storage

4.3.1 Sample extracts must be protected from light and stored at 4°C (±2°C) until 365 days after delivery to the client of a complete data package.

4.3.2 Sample extracts must be stored in an atmosphere demonstrated to be free of all potential contaminants.

4.3.3 Samples, sample extracts, and standards must be stored separately.

4.4 All containers should be glass jars or bottles with Teflon lids. A minimum of one liter is required for aqueous samples. A minimum of 80 g is required for soil samples (125-mL glass jar).

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

5.1 Sources of interference in this method can be grouped into two broad categories: contaminated solvents or reagents. Knowledge of good laboratory practices is assumed, including steps to be followed in routine testing and cleanup of solvents and reagents. The discussion that follows focuses on sources of interference associated with the sample matrix and compound classes that represent common sources of interference, particularly phthalate esters, organosulfur compounds, lipids, and waxes. Interferences co-extracted from the samples will vary considerably from sample to sample. While general cleanup techniques are referenced or provided as part of this method, unique samples may require additional cleanup approaches to achieve desired degrees of discrimination and quantitation.

5.2 Interferences by phthalate esters introduced during sample preparation can pose a major problem in pesticide determinations. These materials may be removed prior to analysis using Gel Permeation Chromatography (GPC) - pesticide option (Method 3640) or as Fraction III of the silica gel cleanup procedure (Method 3630). Common flexible plastics contain varying amounts of phthalate esters that are easily extracted or leached from such materials during laboratory operations. Cross-contamination of clean glassware routinely occurs when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled. Interferences from phthalate esters can best be minimized by avoiding contact with all plastic materials and checking all solvents and reagents for phthalate contamination. Exhaustive cleanup of solvents, reagents and glassware may be required to eliminate background phthalate ester contamination.

5.3 The presence of elemental sulfur will result in large peaks that interfere with the detection of later eluting organochlorine pesticides. Method 3660 is suggested for removal of sulfur.

5.4 Waxes, lipids, and other high molecular weight co-extractables can be removed by GPC.

6.0 EQUIPMENT/APPARATUS

6.1 Separatory funnel - 2 liter, with Teflon stopcock.

6.2 Glass funnel with sodium sulfate.

6.3 Apparatus for grinding dry waste samples.

6.4 Soxhlet extractor – 40 mm ID, with 500 mL round bottom flask.

Drying column – 20 mm ID Pyrex® chromatographic column with Pyrex® glass wool at bottom.

NOTE: Fritted glass discs are difficult to decontaminate after highly contaminated extracts have bee passed through. Columns without frits may be purchased. Use a small pad of Pyrex® glass wool to retain the adsorbent. Pre-wash the glass wool pad with 50 mL of acetone followed by 50 mL of elution solvent prior to packing the column with adsorbent.

6.5 Ultrasonic preparation - A horn type device equipped with a titanium tip, or a device that will give equivalent performance, shall be used.

6.5.1 Ultrasonic Disrupter - The disrupter must have a minimum power wattage of 300 watts, with pulsing capability. A device designed to reduce the cavitation sound is recommended. Follow the manufacturer's instructions for preparing the disrupter for extraction of samples with low and medium/high concentration.

6.5.2 Use a 3/4" horn for the low concentration method and a 1/8" tapered microtip attached to a 1/2" horn for the medium/high concentration method.

6.6 Sonabox or equivalent- Recommended with above disrupters for decreasing cavitation sound (Heat Systems - Ultrasonics, Inc., Model 432B or equivalent).

6.7 Apparatus for determining percent dry weight:

6.7.1 Oven - Drying.

6.7.2 Desiccator.

6.7.3 Crucibles - Porcelain or disposable aluminum.

6.8 Pasteur glass pipettes - 1 mL, disposable.

6.9 Beakers - 400 mL.

6.10 Vacuum or pressure filtration apparatus.

6.10.1 Buchner funnel.

6.10.2 Filter paper - S&S #595 (size 18.5 cm) or equivalent.

6.11 Kuderna-Danish (K-D) apparatus.

6.11.1 Concentrator tube - 10 mL, graduated (Kontes K-570050-1025 or equivalent). A ground-glass stopper is used to prevent evaporation of extracts.

6.11.2 Evaporation flask - 500 mL (Kontes K-570001-500 or equivalent). Attach to concentrator tube with springs or clips.

6.11.3 Snyder column - Three ball macro (Kontes K-503000-0121 or equivalent).

6.11.4 Springs – ½ inch (Kontes K-662750 or equivalent).

6.12 Solvent vapor recovery system (Kontes K-545000-1006 or K547300-0000, Ace Glass 6614-30, or equivalent).

6.13 Glass or paper thimble or glass wool – contaminant free.

6.14 Boiling chips - Solvent extracted and oven baked, approximately 10/40 mesh (silicon carbide or equivalent).

6.15 Water bath - Heated, with concentric ring cover, capable of temperature control (±5°C). The bath should be used in a hood.

6.16 Balance - Top loading, capable of accurately weighing to the nearest 0.01 g to 100g.

6.17 Vials - Glass, 2-mL capacity with Teflon lined screw caps or crimp tops.

6.18 Glass scintillation vials - 20 mL, with aluminum lined screw caps.

6.19 pH indicator paper - narrow range (pH range of 1-6 and 7-11).

6.20 Syringe - 1.0, 2.5, and 10.0 mL.

6.21 Graduated cylinder - 1 liter.

6.22 Spatula - Stainless steel or Teflon.

6.23 Glass funnel.

6.24 Continuous liquid-liquid extractor - Equipped with Teflon or glass connecting joints and stopcocks requiring no lubrication (Kontes 584200-0000, 584500-0000, 583250-0000, or equivalent).

6.25 Heating mantle - Rheostat controlled.

6.26 Erlenmeyer flask – 250 mL

6.27 Automated 8-place Glascol shaker or equivalent.

6.28 Turbovap extract concentrator.

6.29 Test tube rack.

6.30 Pyrex glass wool.

6.31 Volumetric flasks, Class A - 10 mL (optional).

7.0 REAGENTS

7.1 Reagent or pesticide grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of

sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 Organic-free reagent water. All references to water in this method refer to organic-free reagent water.

7.3 Solvents and reagents: As appropriate for Method 3510C, 3520C, 3540C, 3550B, 3630C, 3640A, or 3660B: n-hexane, methylene chloride, acetone, methanol and isooctane (2,2,4-trimethylpentane). All solvents should be pesticide quality or equivalent, and each lot of solvent should be determined to be phthalate free.

7.4 Sodium sulfate (granular, anhydrous), Na_2SO_4. Purify by heating at 400°C for 4 hours in a shallow tray, or by pre-cleaning the Na_2SO_4 with methylene chloride. If the Na_2SO_4 is cleaned with methylene chloride, a method blank must be analyzed, demonstrating that there is no interference from the Na_2SO_4.

7.5 Sulfuric acid solution (1:1 v:v), H_2SO_4. Slowly add 50mL of H_2SO_4 (sp. Gr. 1.84) to 50mL of organic-free reagent water.

7.6 Sodium hydroxide solution, 10 N: (ACS) Dissolve 40 g NaOH in reagent water and dilute to 100 mL.

7.7 All gases (i.e., carrier, auxiliary, etc.) should be at least high purity grade.

7.8 Standards preparation:

Prepare individual stock standard solutions in n-hexane or iso-octane at the following concentrations every <u>six months or sooner</u> if the solution has degraded or concentrated (Assign log numbers to each of the solutions and log them into the Standards Log Book; see Standard Operating Procedure for "Standards Receipt, Traceability, and Preparation", SWL-OP-202).

7.8.1 Stock Standard Solutions - Individual solutions are obtained from AccuStandard or equivalent supplier at concentrations of 1000 µg/mL in hexane.

7.8.1.1 Concentrated and certified standard mixtures can be obtained from several sources. For example, Restek and Supelco supply the pesticide mixtures. It is recommended that these be used for traceability purposes. Stock standard solutions can then be diluted directly from the concentrates.

7.8.1.2 The pesticides can be made in two solutions Individual A (INDA) and Individual B (INDB), or in one solution which has all of the compounds that are in the Individual A and Individual B mixes (INDAB).

7.8.1.3 Stock standard solutions must be kept refrigerated at 4°C (±2°C) for no longer than 6 months.

7.8.2 Calibration Standards — A minimum of five calibration standards for each parameter of interest (except multi-components which use a single point calibration at or below the PQL) should be prepared through dilution of the

highest level standard with hexane or iso-octane. One of the concentrations should be at or below the PQL. The remaining concentrations should bracket the expected range of concentrations found in real samples or should define the working range of the GC/ECD. The components are diluted in hexane or iso-octane. Concentrations are in ng/mL.

7.8.2.1 Prepare solutions of the calibration standards for INDA and INDB or INDAB at 5 concentration levels beginning at the quantitation limit and spanning the linear working range of the detector. See Tables 3 and 4 for the solution concentrations. See Table 5 for the extended list of compounds. Multi-component standard concentrations may be seen in Tables 6 and 7. Table 8 lists the performance evaluation standard concentrations.

7.8.2.2 Calibration standards must be remade at least every six months, or verified against new standards before use.

Table 3
Pesticide Calibration Standards

Individual Standard Mixture A	Low Point Concentration ng/mL (Level 1)
alpha-BHC	3.75
Heptachlor	3.75
gamma-BHC	3.75
Endosulfan I	3.75
Dieldrin	7.5
Endrin	7.5
4,4'-DDD	7.5
4,4'-DDT	7.5
Methoxychlor	37.5
Tetrachloro-m-xylene	3.75
Decachlorobiphenyl	7.5

Table 4

Individual Standard Mixture B	Low Point Concentration ng/mL (Level 1)
beta-BHC	3.75
delta-BHC	3.75
Aldrin	3.75
Heptachlor epoxide	3.75
alpha-Chlordane	3.75
gamma-Chlordane	3.75
4,4'-DDE	7.5
Endosulfan sulfate	7.5
Endrin aldehyde	7.5
Endrin ketone	7.5
Endosulfan II	7.5
Tetrachloro-m-xylene	3.75
Decachlorobiphenyl	7.5

Note: Level 2 concentration = 2 x Level 1 Level 3 concentration = 4 x Level 1
Level 4 concentration = 8 x Level 1 Level 5 concentration = 16 x Level 1

Prepare all solutions in hexane or isooctane. Keep stored at 4°C (±2°C) no longer than 6 months.

Table 5

Individual Standard Mixture AB	Low Point Concentration ng/mL (Level 1)
TCX	3.75
alpha-BHC	3.75
gamma-BHC (Lindane)	3.75
beta-BHC	3.75
delta-BHC	3.75
Heptachlor	3.75
Aldrin	3.75
Heptachlor Epoxide	3.75
gamma-Chlordane	3.75
alpha-Chlordane	3.75
Endosulfan I	3.75
4,4'-DDE	7.5
Dieldrin	7.5
Endrin	7.5
Endosulfan II	7.5
4,4'-DDD	7.5
4,4'-DDT	7.5
Endrin Aldehyde	7.5
Methoxychlor	37.5
Endosulfan Sulfate	7.5
Endrin Ketone	7.5
DCB	3.75

Table 6

Extended List Mixture	Low Point Concentration ng/mL (Level 1)
Chlorobenzilate	37.5
DBCP	TBD
Diallate	72.5
Hexachlorobenzene	TBD
Hexachlorocyclopentadiene	TBD
Isodrin	3.75

TBD = to be determined

Note: Level 2 concentration = 2 x Level 1 Level 3 concentration = 4 x Level 1
Level 4 concentration = 8 x Level 1 Level 5 concentration = 16 x Level 1

Prepare all solutions in hexane or isooctane. Keep stored at 4°C (±2°C) no longer than 6 months.

Table 7

Multi-response calibration standards – Low Point Concentration ng/mL (Level 1)

TOX	Low Point Concentration (ng/mL) (Level 1)
Toxaphene	250

Note: Level 2 concentration = 2 x Level 1 Level 3 concentration = 4 x Level 1
Note: Level 4 concentration = 8 x Level 1 Level 5 concentration = 16 x Level 1

Prepare all solutions in hexane or isooctane. Keep stored at 4°C (±2°C) no longer than 6 months.

Table 8

Multi-response calibration standards – Low Point Concentration ng/mL (Level 1)

T.CHLOR	Low Point Concentration (ng/mL) (Level 1)
Technical Chlordane	10.0

Note: Level 2 concentration = 2 x Level 1 Level 3 concentration = 4 x Level 1
Note: Level 4 concentration = 8 x Level 1 Level 5 concentration = 16 x Level 1

Prepare all solutions in hexane or isooctane. Keep stored at 4°C (±2°C) no longer than 6 months.

Table 9

Performance Evaluation Mixture	(ng/mL)
gamma-BHC	10.0
alpha-BHC	10.0
beta-BHC	10.0
4,4'-DDT	100.0
Endrin	50.0
Methoxychlor	250.0
Tetrachloro-m-xylene	20.0
Decachlorobiphenyl	20.0

Performance Evaluation Mixture (PEM) - prepare the PEM in hexane or isooctane at the concentration levels listed below. The PEM must be prepared every six months or more often if the solution has degraded or concentrated.

7.8.3 Spiking Solutions:

7.8.3.1 Surrogate Solution

The surrogates, Tetrachloro-m-xylene and Decachlorobiphenyl, are added to all standards, samples, matrix spikes, and blanks. Prepare a surrogate spiking solution of 0.2 µg/mL of each of the two compounds in acetone or methanol. The solution should be checked frequently for stability. The solution must be replaced after six months, or sooner, if comparison with quality control check samples indicates a problem.

Caution: Analysts must allow all spiking solutions to equilibrate to room temperature before use.

7.8.3.2 Pesticide Matrix Spiking Solution

Prepare a spiking solution in methanol (acetone produces interfering aldol-condensation products) that contains the pesticides in the concentrations specified in Table 9.

Table 9
Matrix Spiking Solution (µg/mL)

ANALYTE	Concentration (µg/mL)
Aldrin	0.25
Alpha-BHC	0.25
Beta-BHC	0.25
Delta-BHC	0.25
Gamma-BHC	0.25
alpha-Chlordane	0.25
gamma-Chlordane	0.25
4,4'-DDD	0.25
4,4'-DDE	0.25
4,4'-DDT	0.25
Dieldrin	0.25
Endosulfan I	0.25
Endosulfan II	0.25
Endosulfan Sulfate	0.25
Endrin	0.25
Endrin Aldehyde	0.25
Endrin Ketone	0.25
Heptachlor	0.25
Heptachlor Epoxide	0.25
4,4'-Methoxychlor	1.0
Extended List Compounds:	
Chlorobenzilate	1.0
DBCP	TBD
Diallate	2.0
Hexachlorobenzene	TBD
Hexachlorocyclopentadiene	TBD
Isodrin	0.25

7.8.3.2.2 The solution must be prepared every six months or sooner if the solution has degraded or concentrated.

7.8.3.3 Florisil Cartridge Check Solution

7.8.3.3.1 Florisil cleanup is not performed as a routine, but clients may occasionally request its use. Before use, pesticide recoveries are verified through use of the following check solution. Table 10 displays the check solution components and concentrations.

Table 10
Florisil Check Solution (µg/mL)

Analyte	Concentration (µg/mL)
2,4,5-Trichlorophenol	0.10
Alpha-BHC	0.02
Gamma-BHC	0.02
4,4'-DDD	0.04
4,4'-DDT	0.04
Dieldrin	0.04
Endosulfan I	0.04
Endrin	0.04
Heptachlor	0.02
4,4'-Methoxychlor	0.2
TCX	0.02
DCB	0.04

7.8.3.3.2 The check solution should exhibit 80 - 120% recovery of the pesticides and less than 5% recovery of 2,4,5-TCP.

7.8.3.3.3 Store all standard solutions in amber glass bottles or vials with a Teflon-lined screw cap at 4°C (±2°C) and protect from light.

8.0 PROCEDURE

8.1 Extraction Procedure

8.1.1 Upon receiving a copy of the LIMS (Laboratory Information Management System) sample log, the samples are retrieved from storage and logged out for extraction by the technician according to the chain-of-custody procedure.

8.1.2 The technician now initiates the automated extraction logsheet (See Attachment 1 for Extraction Log example). The pertinent extraction information is keyed into the Extraction Laboratory Database, which is updated as the extraction procedure progresses.

8.1.3 After the samples are extracted and are ready for departmental transfer, the database-generated extraction log is reviewed and submitted with the sample extracts to the appropriate laboratory for analysis.

8.1.4 Aqueous Sample Procedure: Aqueous samples may be extracted by the Separatory Funnel method or the Continuous Liquid-Liquid method, depending on the nature and number of the samples. Method 3520C does not recommend Continuous Liquid-Liquid extraction for pesticide samples. Organochlorine pesticides may be dechlorintated by continuous liquid-liquid extraction, thus the Separatory Funnel method is preferred. Continuous Liquid-Liquid extraction may be required if: 1) Emulsions prevent extraction by Separatory Funnel, or 2) Sample Holding times will be missed due to overcapacity of samples in-house. If the Continuous Liquid-Liquid method is used, then all spiking solutions must be made in methanol to prevent aldol-condensation products, which interfere with analyses.

8.1.4.1 Separatory Funnel Extraction (Method 3510C):

8.1.4.1.1 This method is applicable to the isolation and concentration of water-insoluble and slightly water-soluble organics in preparation for a variety of chromatographic procedures.

8.1.4.1.1.1 Using a 1 liter graduated cylinder, measure 1 liter (nominal) of sample and quantitatively transfer it to the separatory funnel. Rinse the graduated cylinder with methylene chloride and pour into the separatory funnel. If high concentrations are anticipated, a smaller volume may be used and then diluted with organic-free

reagent water to 1 liter. The pH of the sample is taken at this time. Pour approximately 10mLs of sample into a scintillation vial, read value, and record in the Extraction Log. Add 1.0 mL of the surrogate standard to all samples, spikes, and blanks and mix well. For the sample in each analytical batch selected for spiking, add 1.0 mL of the matrix-spiking standard and mix well.

8.1.4.1.1.2 After spiking, adjust the pH between 5 and 9, if necessary, using 1:1 sulfuric acid or 10 N sodium hydroxide.

8.1.4.1.1.3 Add 60 mL of methylene chloride to the separatory funnel.

8.1.4.1.1.4 Seal and shake the separatory funnel vigorously for 1-2 minutes with periodic venting to release excess pressure. This process can be performed manually or by an automated 8-place Glascol shaker.

NOTE: Methylene chloride creates excessive pressure very rapidly; therefore, initial venting should be done immediately after the separatory funnel has been sealed and shaken once. The separatory funnel should be vented into a hood to prevent unnecessary exposure of the analyst to solvent vapors.

8.1.4.1.1.5 Allow the organic layer to separate from the water phase for a minimum of 10 minutes. If the emulsion interface between layers is more than one-third the size of the solvent layer, filtration of the emulsion through glass wool, centrifugation, or other physical methods may be required. Collect the solvent extract in an Erlenmeyer flask. If the emulsion cannot be broken (recovery of < 80% of the methylene chloride, corrected for the water solubility of methylene chloride), transfer the sample, solvent, and emulsion into the extraction chamber of a continuous extractor and proceed as described in Method 3520C, Continuous Liquid-Liquid Extraction.

8.1.4.1.1.6 Repeat the extraction two more times using fresh portions of solvent (Sections

8.1.4.1.1.3-8.1.4.1.1.5). Combine the three solvent extracts.

8.1.4.1.1.7 Assemble a Kuderna-Danish (K-D) concentrator (if necessary) by attaching a 10mL concentrator tube to a 500mL evaporation flask.

8.1.4.1.1.8 Dry the extract by passing it through a funnel containing about 100 g of anhydrous sodium sulfate. Collect the dried extract in a K-D concentrator. Rinse the Erlenmeyer flask, which contained the solvent extract, with 20-30 mL of methylene chloride and add it to the column to complete the quantitative transfer.

8.1.4.1.1.9 Add one or two clean boiling chips to the flask and attach a three-ball Snyder column. Prewet the Snyder column by adding about 1 mL of methylene chloride to the top of the column. Place the K-D apparatus on a hot water bath (60-65°C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 45-60 minutes. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume of liquid reaches 10-15mL, allow to cool for approximately 10 minutes.

8.1.4.1.1.10 Rinse the Snyder column into the sample extract, and transfer the extract to a scintillation vial. If the water bath is not to be used, exchange the extract using the nitrogen blowdown apparatus as noted in Section 8.1.4.4.1. If exchange is performed at the water bath, concentrate the extracts to the final volume according to section 8.1.4.4.2.

8.1.4.1.1.11 If sulfur crystals are a problem, proceed to Method 3660 for cleanup. The extract should be adjusted to 10.0 mL with hexane. If GPC is used, concentrate the final extract

to a volume of 5.0 mL to account for the half lost on GPC.

8.1.4.1.1.12 The extract obtained may now be analyzed for analyte content using GC/ECD analysis. Transfer the extract to a vial with a Teflon lined screw-cap or crimp top, and label appropriately.

8.1.4.2 Continuous Liquid-Liquid Extraction (Method 3520C):

8.1.4.2.1 Using a 1-liter graduated cylinder, measure out 1 liter (nominal) of sample and quantitatively transfer it to the continuous extractor. Rinse the graduated cylinder with methylene chloride and pour into the continuous extractor. If high concentrations are anticipated, a smaller volume may be used and then diluted with organic-free reagent water to 1 liter. It is recommended that if high analyte concentrations are anticipated, samples should be collected in smaller sample bottles and the whole sample used. The pH of the sample is taken at this time. Pour approximately 10mLs of sample into a scintillation vial, read value, and record in the Extraction Log. Pipette 1.0 mL of the surrogate standard spiking solution into each sample into the extractor and mix well. For the sample in each analytical batch selected for spiking, add 1.0 mL of the matrix-spiking standard and mix well.

8.1.4.2.2 Add 250 mL of methylene chloride to the liquid-liquid extractor and 200 mL to the distilling flask. Add several boiling chips to the flask.

8.1.4.2.3 Add sufficient water to the extractor to ensure proper operation and extract for 18 to 24 hours.

8.1.4.2.4 Allow to cool, then detach the boiling flask. Dry the extract using the technique referred to in Sections 8.1.4.1.1.8.

8.1.4.2.5 Perform concentration using the Kuderna-Danish (K-D) Technique (Section 8.1.4.3) or Turbovap (proceed according to manufacturer's instructions). Concentrate, if necessary, by following the appropriate steps in Sections 8.1.4.4 through 8.1.4.5.

8.1.4.3 K-D Technique:

8.1.4.3.1 Assemble a Kuderna-Danish (K-D) concentrator by attaching a 10-mL concentrator tube to a 500-mL evaporation flask. Dry the extract by passing it through a glass funnel lined with filter paper and filled with

anhydrous sodium sulfate. Collect the dried extract in a K-D concentrator. Rinse the flask that contained the solvent extract with 20-30 mL of methylene chloride and add it to the column to complete the quantitative transfer.

8.1.4.3.2 Add one or two clean boiling chips to the flask and attach a three-ball Snyder column. Prewet the Snyder column by adding about 1 mL of methylene chloride to the top of the column. Place the K-D apparatus on a hot water bath (15-20°C above the boiling point of the solvent) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed. Adjust the vertical position of the apparatus and the water temperature, as required, to complete the concentration in 45-60 minutes. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume of liquid reaches 10-15 mL, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes. Remove the Snyder column and rinse the flask and its lower joints into the concentrator tube with 1-2 mL of extraction solvent.

8.1.4.3.3 Solvent exchange may be performed at the nitrogen blowdown or at the hot water bath. If exchange is to be performed at the hot-water bath, momentarily remove the Snyder column, add 50 mL of the exchange solvent (hexane), a new boiling chip, and reattach the Snyder column. Concentrate the extract, raising the temperature of the water bath, if necessary, to maintain proper distillation.

8.1.4.4.4 Remove the Snyder column and rinse the flask and its lower joints into the concentrator tube with 1-2 mL exchange solvent (hexane). The extract is then adjusted to a 10.0mL final volume in hexane. Final volumes as low as 1.0mL may be used to achieve lower detection limits, if necessary. Concentration may also be accomplished by the techniques outlined in Sections 8.1.4.4 through 8.1.4.5.

8.1.4.4 Concentration:

Concentration is accomplished by nitrogen blowdown technique and is used to adjust the extract to the final volume required. Solvent exchange is generally performed using the nitrogen blowdown apparatus.

8.1.4.4.1 Nitrogen Blowdown Technique for Solvent Exchange

8.1.4.4.1.1 Place the scintillation vial under the nitrogen stream and evaporate the solvent volume to a final volume of 2.0 mL using a gentle stream of clean, dry nitrogen (filtered through a column of activated carbon).

CAUTION: Plastic tubing must not be used between the carbon trap and the sample, since it may introduce interferences.

8.1.4.4.1.2 Add 10 mL of hexane to the extract and reduce the volume to 2 mL.

8.1.4.4.1.3 Transfer the extract into a 12-mL VOA vial, rinsing the vial with hexane to ensure quantitative transfer, and bring to the desired final volume of 10.0 mL. The extract is brought to a final volume of 5.0 mL if GPC was performed to account for the ½ that was not recovered from the GPC apparatus.

8.1.4.4.2 Nitrogen Blowdown Technique for Concentration if exchange is performed at the hot water bath.

Place the scintillation vial under the nitrogen stream and evaporate the solvent volume to a final volume of 10.0 mL using a gentle stream of clean, dry nitrogen (filtered through a column of activated carbon). The extract is brought to a final volume of 5.0 mL if GPC was performed to account for the ½ that was not recovered from the GPC apparatus.

CAUTION: Plastic tubing must not be used between the carbon trap and the sample, since it may introduce interferences.

8.1.4.5 The extract may now be analyzed for the target analytes. If analysis of the extract will not be performed immediately, store the Teflon-capped VOA vial at 4°C (±2°C) refrigerated. If the extract will be stored longer than 2 days it should be transferred to a vial with Teflon lined screw-caps or crimp tops, and labeled appropriately.

8.1.5 Solid Sample Procedure:

Solid samples may be extracted based on method 3550B, Ultrasonic Extraction, or method 3540C, Soxhlet extraction (The Soxhlet extraction process is only used upon client request or in special instances). The ultrasonic procedure is divided into two sections, based on the expected concentration of organics in the sample. The low concentration method (individual organic components of ≤ 20 mg/kg) uses a larger sample size and a more rigorous extraction procedure (lower

concentrations are more difficult to extract). The medium/high concentration method (individual organic components of > 20 mg/kg) is much simpler and therefore faster. It is highly recommended that the extracts be cleaned up prior to analysis.

8.1.5.1 Sample Handling:

8.1.5.1.1 Sediment/soil samples - Decant and discard any water layer on a sediment sample. Mix sample thoroughly, especially composited samples. Discard any foreign objects such as sticks, leaves, and rocks.

8.1.5.1.2 Determine the dry weight of the sample remaining after decanting. Measurement of soil pH is required.

8.1.5.1.3 Waste samples - Samples consisting of multiphases must be prepared by the phase separation method in Chapter Two of SW-846 before extraction. This procedure is for solids only.

8.1.5.1.4 Dry waste samples amenable to grinding; grind or otherwise subdivide the waste.

8.1.5.1.5 Gummy, fibrous or oily materials not amenable to grinding should be cut, shredded, or otherwise broken up to allow mixing, and maximum exposure of the sample surfaces for extraction. The professional judgment of the analyst is required for handling of these difficult matrices. The addition of anhydrous sodium sulfate to the sample (1:1) may make the mixture amenable to grinding.

8.1.5.1.6 Determination of percent moisture: weigh out a portion of sample for this determination at the same time as the portion used for analytical determination.

WARNING: The drying oven should be contained in a hood or vented. Significant laboratory contamination may result from drying a heavily contaminated hazardous waste sample. However, samples known or suspected to contain significant concentrations of toxic, flammable, or explosive constituents should not be over-dried because of concerns for personal safety. Laboratory discretion is advised. It may be prudent to delay oven-drying of the weighed-out portion until other analytical results are available.

8.1.5.2 Low concentrations of organics and pesticides (≤ 20 mg/kg; Method 3550B):

8.1.5.2.1 The following step should be performed rapidly to avoid loss of the more volatile extractables. Weigh approximately 30g of sample into a 400mL beaker.

Record the weight to the nearest 0.1g (DO NOT TARGET WEIGHTS). Nonporous or wet samples (gummy or clay type) that do not have a free flowing sandy texture must be mixed with approximately 60g of anhydrous sodium sulfate, using a spatula. After addition of sodium sulfate, the sample should be free flowing. Add 1.0mL of pesticide surrogate standards to all samples, spikes, standards, and blanks. For the sample in each analytical batch selected for spiking, add 1.0mL of the pesticide matrix-spiking standard. Immediately add 100mL of 1:1 methylene chloride/acetone.

8.1.5.2.2 Place the bottom surface of the tip of the disrupter horn about ½ inch below the surface of the solvent, but above the sediment layer. Be sure that the horn is properly tuned according the manufacturer's instructions.

8.1.5.2.3 Extract ultrasonically for 3 minutes, with output control knob set at 10 (full power) and with mode switch on Pulse (pulsing energy rather than continuous energy) and percent-duty cycle knob set at 50% (energy on 50% of time and off 50% of time). Do not use microtip probe.

8.1.5.2.4 Decant the extract and filter it through S&S 595 size 18.5 cm filter paper (or equivalent) in a glass funnel filled with sodium sulfate, if needed.

8.1.5.2.5 Repeat the extraction two or more times with two additional 100mL portions of solvent. Decant off the solvent after each ultrasonic extraction. On the final ultrasonic extraction, pour the entire sample into the Buchner funnel and rinse with extraction solvent. Apply a vacuum to the filtration flask, and collect the solvent extract. Continue filtration until all visible solvent is removed from the funnel, but do not attempt to completely dry the sample, as the continued application of a vacuum may result in the loss of some analytes. Alternatively, if centrifugation is used, transfer the entire sample to the centrifuge bottle. Centrifuge at low speed, and then decant the solvent from the bottle.

8.1.5.2.6 Assemble a Kuderna-Danish (K-D) concentrator by attaching a 10mL concentrator tube to a 500mL evaporator flask. Transfer filtered extract to a 500mL evaporator flask and proceed to the next section.

8.1.5.2.7 Add one or two clean boiling chips to the flask and attach a three-ball Snyder column. Prewet the Snyder column by adding about 1mL of methylene chloride to the top of the column. Place the K-D apparatus on a hot water bath (60-

75°C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 45-60 minutes. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume of liquid reaches 10-15mL, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes.

8.1.5.2.8 Rinse and remove the Snyder column, and transfer the extract to a scintillation vial. Exchange (if necessary) and concentrate the extract, using the appropriate nitrogen blowdown technique as outlined in Sections 8.1.4.4 through 8.1.4.5.

8.1.5.2.9 If sulfur crystals are a problem, proceed to Method 3660 for cleanup. The extract should then be adjusted to 10.0 mL with hexane. If GPC is used, concentrate the final extract to a volume of 5.0 mL to account for the half lost on GPC.

8.1.5.2.10 The extract obtained may now be analyzed for analyte content using GC/ECD analysis. Transfer the extract to a vial with Teflon lined screw caps or crimp the top, and label appropriately.

8.1.5.3 Medium/high concentration pesticide/PCB method (individual organic components of > 20 mg/kg):

8.1.5.3.1 Transfer approximately 2g (record weight to the nearest 0.1g) of sample to a 20mL vial. Wipe the mouth of the vial with a tissue to remove any sample material. Record the exact weight of sample taken. Cap the vial before proceeding with the next sample to avoid any cross contamination.

8.1.5.3.2 Add 2g of anhydrous sodium sulfate to sample in the 20mL vial and mix well.

8.1.5.3.3 Surrogate standards are added to all samples, spikes, and. Add 1.0mL of pesticide surrogate spiking solution to sample mixture. For the sample in each analytical batch selected for spiking, add 1.0mL of the pesticide matrix-spiking standard.

8.1.5.3.4 Immediately add whatever volume of solvent is necessary to bring the final volume to 10.0mL considering the added volume of surrogates and matrix spikes. Disrupt the

sample with the 1/8 inch tapered microtip ultrasonic probe for 2 minutes at output control setting 5 and with mode switch on pulse and percent duty cycle at 50%. The extraction solvent for organochlorine pesticides is hexane.

8.1.5.3.5 Loosely pack disposable Pasteur pipettes with 2 to 3cm Pyrex glass wool plugs. Filter the extract through the glass wool and collect 5.0mL in a concentrator tube if further concentration is required. See previous section for details on extract concentration.

8.1.5.3.6 The extract is ready for cleanup or analysis, depending on the extent of interfering co-extractives.

8.1.5.4 Soxhlet Extraction (Method 3540C):

8.1.5.4.1 Immediately after weighing the sample for extraction, weigh 5-10g of the sample into a disposable aluminum weighing boat. Determine the % moisture of the sample by drying at 103°C (±2°C) for a predetermined amount of time or over night. Allow to cool before weighing. See Section 9.0, "Calculations" for calculation of percent moisture.

8.1.5.4.2 Blend 30g of the solid sample with 30g of anhydrous sodium sulfate and place in an extraction thimble. The extraction thimble must drain freely for the duration of the extraction period. A glass wool plug above and below the sample in the Soxhlet extractor is an acceptable alternative for the thimble. Add 1.0mL of the surrogate standard spiking solution onto the sample. For the sample in each analytical batch selected for spiking, add 1.0mL of the matrix-spiking standard.

8.1.5.4.3 Place approximately 400mL of the extraction solvent (methylene chloride: acetone, 1:1 v/v) into a 500mL round bottom flask containing one or two clean boiling chips.

Attach the flask to the extractor and extract the sample for 18 hours at 4-6 cycles/hr.

8.1.5.4.4 Allow the extract to cool after the extraction is complete.

8.1.5.4.5 Perform concentration (if necessary) using the Kuderna-Danish (K-D) Technique (Section 8.1.4.3) or Turbovap (proceed according to manufacturer's instructions). See Sections 8.1.4.4 through 8.1.4.5 for exchange and/or extract concentration.

8.1.5.5 Waste Dilution

8.1.5.5.1 This method describes a solvent dilution of a non-aqueous waste sample prior to cleanup and/or analysis. It is designed for wastes that may contain organic chemicals at a concentration greater than 20,000 mg/Kg and that are soluble in the dilution solvent.

8.1.5.5.2 Samples consisting of multiphases must be prepared by the phase separation method before extraction.

8.1.5.5.3 The sample dilution may be performed in a 10-mL volumetric flask. If disposable glassware is preferred, the 20-mL scintillation vial may be calibrated for use. Pipette 10.0 mL of extraction solvent into the scintillation vial and mark the bottom of the meniscus. Discard this solvent.

8.1.5.5.4 Transfer approximately 1 g of each phase of the sample to separate 20mL vials or 10-mL volumetric flasks (record weight to the nearest 0.1 g). Wipe the mouth of the vial with a tissue to remove any sample material. Cap the vial before proceeding with the next sample to avoid any cross-contamination.

8.1.5.5.5 Add 2.0mL surrogate spiking solution to all samples and blanks. For the sample in each analytical batch selected for spiking, add 2.0mL of the matrix-spiking standard.

8.1.5.5.6 Immediately dilute to 10mL with the methylene chloride.

8.1.5.5.7 Add 2.0g of anhydrous sodium sulfate to the sample.

8.1.5.5.8 Cap and shake the sample for 2 min.

8.1.5.5.9 Loosely pack disposable Pasteur pipettes with 2-3cm glass wool plugs. Filter the extract through the glass wool and collect 5mL of the extract in a tube or vial.

8.1.5.5.10 The extract is exchanged to hexane and is then ready for cleanup or analysis, depending on the extent of interfering co-extractives.

8.1.5.5.11 Any reagent blanks and matrix spike samples must be subjected to exactly the same analytical procedures as those used on actual samples.

9.0 CALCULATIONS

Not applicable.

10.0 QUALITY ASSURANCE/QUALITY CONTROL

10.1 Refer to Chapter One of SW-846 for specific quality control (QC) procedures. Quality control to validate sample extraction is covered in Method 3500 and in the extraction method utilized. If extract cleanup was performed, follow the QC in Method 3600 and in the specific cleanup method.

10.2 Calculate surrogate standard recovery on all samples, blanks, and spikes. Determine if the recovery is within limits established. If recovery is not within limits, the following are required:

10.2.1 Check to be sure there are no errors in calculations and surrogate solutions. Also, check instrument performance.

10.2.2 Recalculate the data and/or reanalyze the extract if any of the above checks reveal a problem.

10.2.3 If both method blank surrogate recoveries are outside of control limits, reanalyze the sample to rule out instrument problems. Re-extract and reanalyze the samples if none of the above are a problem.

10.3 If greater than 50% of the spike recoveries are outside of laboratory control limits in both the LCS and LCSD or any spike recovery is below 10%, the LCS/LCSD and associated samples must be re-extracted and reanalyzed. If out-of-control analytes are out high and those analytes are not present in the samples, then no corrective action is required. Control limits will be those annually updated from in-house generated data. See "Blue Book" for SWLO's current control limits.

10.4 If spike recoveries in the MS/MSD are outside of control limits but the combined LCS/LCSD are within control limits, the poor MS/MSD recoveries may be attributed to matrix effect. If both LCS/LCSD and MS/MSD have greater than 50% of the spiked compounds outside of control limits, the samples and associated QC must be re-extracted and reanalyzed. Control limits will be those annually updated from in-house generated data. See "Blue Book" for SWLO's current control limits.

11.0 WASTE DISPOSAL / POLLUTION PREVENTION

See Standard Operating Procedure SWL-GA-114, "*Hazardous Waste Management Plan.*"

12.0 METHOD PERFORMANCE

12.1 The MDL concentrations are updated annually and are obtained using organic-free reagent water and sandy loam soil. MDL results are maintained in SWLO's "Red book" by the QA Department. Details for determining MDLs are given in Chapter One of SW-846 and the SOP for the "*Definition and Determination of Detection Limits*", SWL-GA-113. The MDL actually achievable in a given analysis will vary depending on detector response characteristics, irreducible noise from instrument electronics, and matrix effects.

12.2 The accuracy and precision obtainable following this method will be determined by the sample matrix, sample preparation technique, optional cleanup techniques, and calibration procedures used.

12.3 The laboratory should maintain control charts of LCS, MS, and MSD recoveries and relative percent deviations between MS and MSD recovery for accuracy and precision data. Surrogate recovery is also plotted. The control charts are monitored monthly for LCS and quarterly for MS and are maintained on file in the laboratory. Control limits are updated on an annual basis (See SOP "*Control Charts*", SWL-GA-112 for more details). See "Blue Book" for SWLO's most current control limits.

12.4 Examples of the separations on a pesticide column are shown in Attachments 2-5. QLs are calculated by multiplying the MDL value for an analyte by a number ranging from 5-10. The QL will, in no instance, be less than the MDL. See "Red Book" for SWLO's QLs.

13.0 REFERENCES

U.S.EPA SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods: "Method 3500B. Organic Extraction and Sample Preparation" Revision 2, December 1996.

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Standard Operating Procedure, "Standards Receipt, Traceability, and Preparation", SWL-OP-202.

Standard Operating Procedure, "Laboratory Safety Plan", SWL-GA-111.

Standard Operating Procedure, "Control Charts", SWL-GA-112.

Standard Operating Procedure, "Definition and Determination of Detection Limits", SWL-GA-113.

Standard Operating Procedure, "Hazardous Waste Management Plan", SWL-GA-114.

Headquarters Air Force Center for Environmental Excellence Quality Assurance Project Plan, Version 2.0, January 1997.

U.S.EPA, Statement of Work OLM03.2, "Organic Analysis: Multi-media, Multi-concentration".

14.0 DEFINITIONS

N/A:..... not applicable

MSDS: ..... Material Safety Data Sheet: Information provided by commercial vendors for the chemical products prepared by their company. The information on the MSDS includes but is not limited to composition, physical properties, hazard identification, first aid measures, emergency cleanup and/or response, exposure, and storage.

SDG: ..... The Sample Delivery Group; a unit within a single case that is used to identify a group of samples for delivery. A SDG is a group of 20 or fewer field samples within a case, received over a period of up to 14 calendar days (7 calendar days for 14-day turns). Data from all samples in a SDG are due concurrently.

15.0 ATTACHMENTS

Attachment 1: Sample Extraction Log

ATTACHMENT 1
Sample Extraction Log

(3520C) SEP-F		
(3510C)	XXXXX	
SHAKER		
SONC. (3550B)		
SOXHLET		

BATCH: 000829WC

EXTRACT DATE 2000-08-29 12:29

001737

SAMPLE ID (EPISODE #)	CLIENT CASE	DATE RECEIVED CLIENT SAMPLE ID	MATRIX/LEVEL	SAMPLE pH	% MOISTURE	DRY WEIGHT	AMOUNT EXTRACTED	EXTRACTED VOLUME	GPC VOLUME	GPC ELUTE VOL	FLORISIL VOL CONC	FLORISIL FIN VOL CONC	VOL SV (P)	FIN PROCESS VOLUME	SCREEN Y/N	MS SV (P)	SURR SV (P)	BASE pH CONFIRMATION	ACID pH CONFIRMATION
					%	g	g or ml	ml	ml	ml	ml	ml	ml	ml	Y/N	µL	µL		Bottle
BL0829WC			W/L				1000	300					10.0	10.0	N		1000		
BB0829WC			W/L				1000	300					10.0	10.0	N		1000		
LC0829WC			W/L				1000	300					10.0	10.0	N	1000	1000		
LD0829WC			W/L				1000	300					10.0	10.0	N	1000	1000		
44097.04 GC801	BECJACOB	08/25/2000 10:45 SA109	W/L	8.08			1000	300					10.0	10.0	N		1000		A
44097.09 GC801	BECJACOB	08/25/2000 10:45 SA123	W/L	7.41			1000	300					10.0	10.0	N		1000		AB
44097.14 GC801	BECJACOB	08/25/2000 10:45 SA156	W/L	7.55			1000	300					10.0	10.0	N		1000		AB
44097.19 GC801	BECJACOB	08/25/2000 10:45 SA137	W/L	7.78			1000	300					10.0	10.0	N		1000		AB
44097.24 GC801	BECJACOB	08/25/2000 10:45 SA79	W/L	8.17			1000	300					10.0	10.0	N		1000		AB
44097.29 GC801	BECJACOB	08/25/2000 10:45 SA54	W/L	7.75			1000	300					10.0	10.0	N		1000		AB

INITIALS

WEIGHING:	JM	EXTRACTION:	JM	FLORISIL:	MeCl2 Hexane
CLEANUP:		KD:	JM		
BLOWDOWN:	JM	TV:			
SURROG. SPIKER:	JM	VERIFIER:	GS		
MATRIX SPIKER:	JM	VERIFIER:	GS		
SUBMITTED BY:	JM	REVIEWED BY:			

ID NUMBERS

FLORISIL CARTRIDGE #:

GPC COLUMN #:

SURROGATES ID CONC #: 5-218-5

MATRIX SPIKE SOLID/CONC #(s): 5-572-4

SOP - METHOD # : SWL-OP- 414 Rev. 2.1

COMMENTS

Pg 29 of 1 Rear

CONT EX. (3520C)	
SEP-F (3510C)	
SHAKER	
SONC. (3550B)	XXXXX
SOXHLET	

BATCH: 000901SC

EXTRACT DATE: 2000-09-01 12:44

001755
Page: 1 of 1

SAMPLE ID (EPISODE #)	CLIENT CASE	DATE RECEIVED / CLIENT SAMPLE ID	MATRIX/LEVEL	SAMPLE pH	% MOISTURE	DRY WEIGHT	AMOUNT EXTRACTED	EXTRACTED VOLUME	GPC VOLUME	GPC ELUTE VOL	FLORISIL VOL CONC	FLORISIL VOL CONC	VOL SV (P)	FIN PROCESS VOLUME	SCREEN Y/N	MS SV (P)	SURR SV (P)	BASE pH CONFIRMATION	ACID pH CONFIRMATION	
					%	g	g (ml)	ml	ml	ml	ml	ml	ml	ml	Y/N	µL	µL			Bottle
BL0901SC			S/L				30.02	30.00			2.0	2.0		10.0	N		1000			
BB0901SC			S/L				29.98	30.0						10.0	N		1000			
LC0901SC			S/L				30.01	30.0						10.0	N	1000	1000			
LD0901SC			S/L				31.01	30.0						10.0	N	1000	1000			
44132.04 GC801	ENSAFE-D	08/30/2000 09:50 CEI-B03-1	S/L	7.79	5.04	28.4	29.94	30.00						10.0	N		1000			A
44132.04MS GC801	ENSAFE-D	08/30/2000 09:50 CEI-B03-1	S/L	7.79	5.04	28.5	29.97	30.00						10.0	N	1000	1000			A
44132.04MSD GC801	ENSAFE-D	08/30/2000 09:50 CEI-B03-1	S/L	7.79	5.04	28.5	30.04	30.00			2.0	2.0		10.0	N	1000	1000			A

S/Gel 5/gel

INITIALS

WEIGHING:		EXTRACTION:	SR	FLORISIL:	
CLEANUP:		KD:	JW		MeCl2
BLOWDOWN:	RKM	TV:	CR		Acetone
SURROG. SPIKER:	SR	VERIFIER:	GS		
MATRIX SPIKER:	SR	VERIFIER:	GS		
SUBMITTED BY:	RKM	REVIEWED BY:			9/5/2000

ID NUMBERS

FLORISIL CARTRIDGE #:

GPC COLUMN #:

SURROGATES ID CONC #: 5-218-5

MATRIX SPIKE SOLID/CONC #(s): 5-572-4

SOP - METHOD # : SWL-OP- 414 Rev. 2.1

COMMENTS

S/Gel by Chris 9/5/2000

Pg 29 Bk 1 29

SOUTHWEST LABORATORY OF OKLAHOMA, INC. *and*
AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.

TM

Standard Operating Procedure

Preparation of Nitroaromatics and Nitramines by HPLC and Nitroglycerin by HPLC SW846 Method 8332

Document No.: SWL-OE-410

Rev No. / Date: Rev. 2.0 — 03/06/01

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APPROVALS	DATES
Procedure Prepared By	3/7/2001 Date
Program Manager or Section Supervisor	3-8-01 Date
Laboratory Safety Officer	3/12/2001 Date
Laboratory QA/QC Officer	3/9/01 Date

(Effective Date is 5 calendar days after the last signature above - QA/QC Officer)

Document Status

[ ] ~~Controlled~~ 05/01/01 DC No. _____ Issued to: _____ Date: _____ QA: _____

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PREPARATION OF NITROAROMATICS AND NITRAMINES BY HPLC AND NITROGLYCERIN BY HPLC SW846 METHOD 8332

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1.0 SCOPE AND APPLICATION

1.1 This method is used to determine the concentration of the following explosive residues and nitroglycerin in water, soil, or sediment. Test codes encompassed by this method includes GC200, GC201, GC202, GC218, and GC299. The method can be used to determine analyte concentrations in the parts per billion (ppb) range.

Table 1

COMPOUND	ABBR.	CAS Number
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine	HMX	2691-41-0
Hexahydro-1,3,5-trinitro-1,3,5-triazine	RDX	121-82-4
1,3,5-Trinitrobenzene	TNB	99-35-4
1,3-Dinitrobenzene	DNB	99-65-4-0
Methyl-2,4,6-trinitrophenylnitramine	Tetryl	479-45-8
Nitrobenzene	NB	98-95-3
2,4,6-Trinitrotoluene	TNT	18-96-7
4-amino-2,6-dinitrotoluene	4ADNT	1946-51-0
2-amino-4,6-dinitrotoluene	2ADNT	35572-78-2
2,6-Dinitrotoluene	26DNT	606-20-2
2,4-Dinitrotoluene	24DNT	121-14-2
o-Nitrotoluene	2NT	88-72-2
p-Nitrotoluene	4NT	99-99-0
m-Nitrotoluene	3NT	99-08-1
SURROGATE		
3,4-Dinitrotouene	34DNT	610-39-9
* Chemical Abstracts Service Registry number		

Table 2
Extended Target List

COMPOUND	ABBR.	CAS Number
Pentaerythritol tetranitrate	PETN	75-11-5

NOTE: See Section 8.1 for required modifications to chromatographic conditions.

1.2 This SOP is based on SW846, Update III, Dec. 1997 method 8330, Rev. 0, Sept. 1994 with no modifications.

1.3 All of these compounds are used in the manufacturing of explosives or are the degradation products of compounds used for that purpose. When making stock solutions for calibration, treat each compound as if it were extremely explosive.

1.4 The practical quantitation limits (PQLs) of target analytes are presented in Table 3. The PQLs are based on the low point (L1) in the initial calibration represented in Table 4, the amount of sample extracted, and the volume of the extract.

1.5 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 explosive materials. Each analyst must demonstrate the ability to generate acceptable results with this method.

1.6 Method Detection Limits (MDLs) are generated according to SWL-GA-113 (see "Red Book" for current MDLs.)

Table 3
Practical Quantitation Limits

Compound	Low Level Water (ug/L)	High Level Water (ug/L)	Soil (ug/Kg)
HMX	0.65	100	500
RDX	0.65	100	500
TNB	0.65	100	500
DNB	0.65	100	500
TETRYL	0.65	100	500
NB	0.65	100	500
TNT	0.65	100	500
24DNT	0.65	100	500
26DNT	0.65	100	500
2NT	0.65	100	500
3NT	0.65	100	500
4NT	0.65	100	500
2ADNT	0.65	100	500
4ADNT	0.65	100	500

Table 4
Calibration Standards (µg/L)

Compound	L1	L2	L3	L4	L5
HMX	0.05	0.2	0.8	3.2	12.8
RDX	0.05	0.2	0.8	3.2	12.8
TNB	0.05	0.2	0.8	3.2	12.8
DNB	0.05	0.2	0.8	3.2	12.8
TETRYL	0.05	0.2	0.8	3.2	12.8
NB	0.05	0.2	0.8	3.2	12.8
TNT	0.05	0.2	0.8	3.2	12.8
4ADNT	0.05	0.2	0.8	3.2	12.8
2ADNT	0.05	0.2	0.8	3.2	12.8
26DNT	0.05	0.2	0.8	3.2	12.8
24DNT	0.05	0.2	0.8	3.2	12.8
2NT	0.05	0.2	0.8	3.2	12.8
4NT	0.05	0.2	0.8	3.2	12.8
3NT	0.05	0.2	0.8	3.2	12.8
34DNT (S)	0.05	0.2	0.8	3.2	12.8

Table 5
Calibration Standards (ug/L)

Compound	CAS #	L1	L2	L3	L4	L5
Nitroglycerin	55-63-0	0.0500	0.200	0.800	3.20	12.8
1,2-DNB (Surrogate)	528-29-0	0.0250	0.100	0.400	1.60	6.40

2.0 METHOD SUMMARY

2.1 Aqueous Samples:

2.1.1 High level water (Direct Injection Method)

Samples are diluted 2X (v/v) with acetonitrile, separated on a Carbosorb reverse phase column (or equivalent), determined at 254nm, and confirmed on a Zorbax SB-CN column (or equivalent).

2.1.2 Low level water (Salting out):

The samples are extracted using NaCl and acetonitrile. The extracts are diluted 2X (v/v) with 0.5 % calcium chloride/water, separated on a Carbosorb reverse phase column (or equivalent), determined at 254 nm, and confirmed on a Zorbax SB-CN column (or equivalent).

2.2 Soil and sediment samples are extracted using acetonitrile in an ultrasonic bath. The extracts are filtered and diluted 2X (v/v) with 0.5 % calcium chloride/water.

3.0 HEALTH AND SAFETY

3.1 Standard precautionary measures used for handling other toxic organic compounds should be employed for safe handling of the analytes targeted by Method 8330.

3.2 Refer to SWL-GA-111, "*Laboratory Safety Plan*".

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

4.1 Grab samples must be collected and stored in glass containers. Follow conventional sampling procedures.

4.2 Samples must be stored in the dark at 4 ± 2°C from the time of collection prior to analysis. Soil or sediment samples must be extracted within 14 days of the collection, and analyzed within 40 days of extraction. Water samples must be extracted within 7 days of collection, and analyzed within 40 days of extraction. High concentration water samples must be analyzed within 14 days of extraction

4.3 Soil and sediment samples after collection should be air dried at room temperature or colder to constant weight. While it is possible to analyze wet soil samples, it is much more difficult to obtain a homogeneous subsample on a wet sample. If wet soil samples are to be analyzed, a moisture determination must be made on a separate subsample.

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

5.1 Tetryl decomposes rapidly in methanol/water solutions, as well as with heat and light. All aqueous samples expected to contain Tetryl should be diluted with acetonitrile prior to filtration. All samples expected to contain Tetryl should not be exposed to temperatures above room temperature.

5.2 Degradation products of Tetryl appear as a shoulder to the TNT peak.

5.3 Salt present in acetonitrile extracted in the last step of the low concentration water (salting out) procedure produces interference peaks at the beginning of the chromatogram.

6.0 EQUIPMENT/ APPARATUS

6.1 HPLC System

6.1.1 Autosampler

6.1.2 Pump - high-pressure pump capable of 6000 psi at 10 ml/min.

6.1.3 Column - Carbosorb, 5um, 250mm x 4.6mm ID (or equivalent) for quantitation
Zorbax SB-CN, 5um, 250mm x 4.6mm ID (or equivalent) for confirmation

6.1.4 Column Heater

6.1.5 Detector - Variable UV. Programmable UV is required when PETN is added to the target list.

6.1.6 Data System - Hewlett Packard Enviroquant.

6.2 Materials

6.2.1 Filters – Gelman Accuspec CR 0.45 µm filters for filtering extracts, or equivalent

6.2.2 Glass Pipettes – Class A, 10mL, 5mL, 4mL, 2mL, and 1mL.

6.2.3 Glass Vials – 1.5 mL, 12mL, 40ml, Teflon lined screw cap or equivalent.

6.2.4 Volumetric flasks – Class A - 10mL, 20mL, 50mL, 100mL, 200mL, 250mL

6.2.5 Rainin Pipettor – 10-100 µL, 100–1000 µL, and 1-10mL.

7.0 REAGENTS

7.1 HPLC grade chemicals shall be used in all tests. 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. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lowering the accuracy of the determination.

7.1.1 Acetonitrile, CH3CN – HPLC grade.

7.1.2 Methanol, CH3OH – HPLC grade.

7.1.3 Water, H2O – Millipore (or equivalent).

7.2 Stock Standard Solutions. Certified standard mixes obtained from a vendor that contain the target analytes.

7.3 Working Calibration Standards.

Prepare 5 working calibration standards by diluting the stock standards with 50:50 (v/v) with a 5 g/L (0.5%) Calcium Chloride/water and acetonitrile. The low standard is at the

PQL. Calibration verifications will be tested daily against the 5 point calibration. As long as the response has not deteriorated, the working calibration standards may be used up to 6 months.

7.4 LCS (Laboratory Control Spike) and LCSD (Laboratory Control Spike Duplicate)

LCS are spiked with the analytes of interest at the midpoint level of the calibration curve.

7.5 MS (Matrix Spike) and MSD (Matrix Spike Duplicate)

MS and MSD are spiked with the analytes of interest at the midpoint level of the calibration curve.

7.6 Surrogate

34DNT, the analyte of non-interest used for the surrogate, is spiked at the midpoint level of the calibration curve.

7.7 Eluent

7.7.1 To prepare 1 liter 50% Methanol/H2O for the mobile phase, add 500ml of methanol to 500mL of HPLC water.

7.7.2 To prepare 1 liter of 50% Acetonitrile/H2O, add 500ml of Acetonitrile to 500ml of HPLC water.

7.7.3 The eluent should be filtered through a 0.45 µm filter subsequent to use.

7.7.4 The eluent should have helium continuously bubbled through the solution in order to remove air.

7.7.5 Prepare 0.5% calcium chloride/water by adding 5g of the solute to 1000 mL of HPLC water. Stir the solution on the stirring plate until all of the Calcium chloride has been dissolved. Place the solution in the refrigerator at 4°C if the solution is not used.

8.0 PROCEDURES

8.1 Procedure for GC200 (Explosives)

8.1.1 Aqueous Samples — High water aqueous samples (Also GC202, See section 8.3)

8.1.1.1 Acrodisc 5mls sample into 10mL amber vials.

8.1.1.2 Add 4.75ml acetonitrile to all samples and blanks and 4.50ml acetonitrile to LC/LD and MS/MSD.

8.1.1.3 Add 250uL of explosive surrogate to all samples, blanks, LC's and MS/MSD.

8.1.1.4 Add 250uL of explosive matrix spike to the LC/LD and MS/MSD.

8.1.1.5 Vortex all samples and blanks for approximately 10 seconds.

8.1.1.6 Complete extraction sheet for the batch using 5mLs as amount extracted and 10mLs as final volume.

8.1.2 Soil Samples (Also GC201, GC299)

8.1.2.1 Weigh approximately 2gm soil sample (previously air dried and ground to a fine consistency) into a pre-clean 40mL voa vial and 2.0gm of pre-extracted clean sand for blanks and LC/LD respectively in voa vials also.

8.1.2.2 Add 500uL explosive surrogate to all samples including blanks.

8.1.2.3 Add 500uL matrix spike solution to the LC/LD and MS/MSD respectively.

8.1.2.4 Add 9.5mL acetonitrile to all blanks and samples and 9.0mLs to the LC/LD and MS/MSD.

8.1.2.5 Sonicate all samples for 18 hours in a water bath. Samples must be covered to prevent photodecomposition.

8.1.2.6 Prepare two (2) sets of clean 12mL clear vials for each sample. To the first set, add 5.0mL of 0.5% calcium chloride solution, then add 5.0mLs of the sonicated sample to each corresponding vial.

8.1.2.7 Vortex each sample for 10 seconds and then centrifuge for 10 minutes (5000 rpm.)

8.1.2.8 Acrodisc (filter) each sample in the second corresponding labeled vial (12mL.)

8.1.2.9 Complete extraction sheet for batch indicating 2gm as amount extracted and 20mL final volume.

8.2 Procedure for GC218 —Low water (salting out) aqueous samples

8.2.1 Prior to analysis, dilute each extract with HPLC water, then shake thoroughly. The samples are ready for analysis.

8.2.2 Add 251.3g of Sodium Chloride to a 1-liter volumetric flask. Measure out 770mL of a water sample in a 1000mL-graduated cylinder.

8.2.3 Transfer part of the sample to a volumetric flask. Gently swivel the flask, until part of the salt has been dissolved. Add the rest of the sample and stir bat to the flask. Mix at maximum speed on a magnetic stirrer until the salt is completely dissolved.

8.2.4 Add 164mL of acetonitrile (measured with a 250mL-graduated cylinder) while the solution is being stirred. Stir for an additional 15 minutes. Turn off the stirrer and allow the phrase to separate for 10 minutes.

8.2.5 Remove the acetonitrile (upper) layer (about 8mL) with a Pasteur pipette and transfer it to a 100mL volumetric flask. Add 10mL of fresh acetonitrile to the water sample in the 1.0-liter flask. Stir the content of the flask for 15 minutes followed by 10 minutes for phase separation. Combine the second acetonitrile portion with the initial extract. The inclusion of a few drops of salt water at this point is not important.

8.2.6 Prepare 32.5% of sodium chloride according to the number of samples (325g of NaCl dissolved in to 1000mL of Millipore water.)

8.2.7 Add 84mL of 32.5% sodium chloride (8.1.2.6) to the acetonitrile from the previous step. Add a stir bar and stir the contents on a magnetic stirrer for 15 minutes followed by 10 minutes for phase separation. Carefully transfer the acetonitrile phase to a 12.0mL vial with Teflon lined screw cap. At this stage, the amount of water transferred with the acetonitrile must be minimized. The water contains a high concentration of NaCl which produces an interfering peak.

8.2.8 Add an additional 1mL of acetonitrile to the 100mL volumetric flask. Again, stir the contents of the flask for 15-minutes followed by 10 minutes for phase separation. Combine the second acetonitrile portion with the initial extract. The final volume should be 5.0mL.

8.2.9 If the extract volume exceeds the requirement, very gently blow down the extract with a minute stream of nitrogen, until the desired volume is achieved.

8.2.10 If the diluted extract is turbid, filter it through a 0.45um Teflon filter using a disposable syringe. Discard the first 0.5mL of filtrate, and retain the remainder in a glass vial with a Teflon lined screw cap.

8.2.11 Prior to analysis, dilute the extracts 2X (v/v) with HPLC water or equivalent. If the extracts are not analyzed immediately, store them in the refrigerator at 4° C until analysis.

8.3 Procedure for GC202 (Nitroglycerin)

8.3.1 Aqueous Samples

8.3.1.1 Acrodisc 5mLs sample into 10mL amber vials.

8.3.1.2 Add 4.75mL acetonitrile to all samples and blanks and 4.50mL acetonitrile to LC/LD and MS/MSD.

8.3.1.3 Add 250uL of explosive surrogate to all samples, blanks, LC's, and MS/MSD.

8.3.1.4 Add 250uL of explosive matrix spike to the LC/LD and MS/MSD.

8.3.1.5 Vortex all samples and blanks for approximately 10 seconds.

8.3.1.6 Complete extraction sheet for the batch using 5mLs as amount extracted and 10mLs as final volume.

8.3.2 Soil Samples

8.3.2.1 Weigh approximately 2gm soil sample (previously air dried and ground to a fine consistency) into a pre-clean 40mL voa vial and 2.0gm of pre-extracted clean sand for blanks and LC/LD respectively in voa vials also.

8.3.2.2 Add 500uL explosive surrogate to all samples including blanks.

8.3.2.3 Add 500uL matrix spike solution to the LC/LD and MS/MSD respectively.

8.3.2.4 Add 9.5mL acetonitrile to all blanks and samples and 9.0mLs to the LC/LD and MS/MSD.

8.3.2.5 Sonicate all samples for 2 hours in a water bath. Samples must be covered to prevent photodecomposition.

8.3.2.6 Prepare two (2) sets of clean 12mL clear vials for each sample. To the first set add 5.0mL of 0.5% calcium chloride solution, then add 5.0mLs of the sonicated sample to each corresponding vial.

8.3.2.7 Vortex each sample for 10 seconds and then centrifuge for 10 minutes (5000rpm.)

8.3.2.8 Acrodisc (filter) each sample in the second corresponding labeled vial (12mL.)

8.3.2.9 Complete extraction sheet for batch indicating 2gm as amount extracted and 20mL final volume.

9.0 CALCULATIONS

Not Applicable.

10.0 QUALTITY ASSURANCE/QUALITY CONTROL

10.1 Prior to preparation of stock solutions, acetonitrile, methanol, and water blanks should be analyzed to determine possible interferences with analyte peaks. If the acetonitrile, methanol, or water blanks show contamination, a different batch should be used.

10.2 Method Blanks

10.2.1 Method blanks for the analysis of aqueous samples should be organic-free reagent water carried through all sample storage and handling procedures.

10.2.2 Method blanks for the analysis of soil samples should be uncontaminated soil carried through all sample storage, extraction, and handling procedures.

10.3 Laboratory Control Spikes

See "Blue Book" for current laboratory control limits.

10.4 Matrix Spikes

10.4.1 See "Blue Book" for current laboratory control limits.

10.4.2 Surrogate — To monitor extraction and analytical performance, all of the extracts are spiked with a surrogate.

11.0 WASTE DISPOSAL/POLLUTION PREVENTION

See Standard Operating Procedure "*Hazardous Waste Management Plan*", SWL-GA-114.

12.0 METHOD PERFORMANCE

The efficiency of the extraction process is monitored through the analytical results of the Method Blank, Laboratory Control Spike, Matrix Spike and Surrogate Recoveries.

13.0 REFERENCES

13.1 Bauer, C.F., S.M. Koza, and T.F. Jenkins, "Collaborative Test Results for a Liquid Chromatographic Method for the Determination of Explosives Residues in Soil," manuscript submitted to the Journal of the AOAC, April 1989.

13.2 Department of the Army, "Reversed-Phase HPLC Method for the Determination of Explosive Residues in Soil," Appendix B, provided by Dennis J. Wynne, Chief, Technology Division, U.S. Army Toxic and Hazardous Materials Agency, Aberdeen Proving Ground, Maryland 21010-5401.

13.3 Department of the Army, "An Improved RP-HPLC Method for the Determination of Nitroaromatics and Nitroamines in Water" Appendix B, provided by Dennis J. Wynne, Chief, Technology Division, U.S. Army Toxic and Hazardous Materials Agency, Aberdeen Proving Ground, Maryland 21010-5401.

13.4 SW846 — Update III, Dec. 1997, Method 8330, Rev. 0, Sept. 1994.

14.0 DEFINITIONS

HPLC: .....High Performance Liquid Chromatograph

LCS: .....Laboratory Control Spike

MS:.....Matrix Spike

15.0 ATTACHMENTS

15.1 Attachment 1a: Sample Extraction Log — Explosives (GC200 — Water)
Attachment 1b: Sample Extraction Log — Explosives (GC200 — Soil)
Attachment 1c: Sample Extraction Log — Explosives (GC201 — Soil)
Attachment 1d: Sample Extraction Log — Explosives (GC299 — Soil)

15.2 Attachment 2: Sample Extraction Log — Low Level Water (GC218 — Soil)

15.3 Attachment 3: Sample Extraction Log — Nitroglycerin (GC202 — Soil)

ATTACHMENT 1a
Sample Extraction Log — Explosives (GC200 — Water)

BATCH

TCLP(C)			
SHAKER			
SONC. (3550B)			XXXXX
SOXHLET			

EXTRACT DATE 00-12-12-15:47

002166
Page: 1 of 1

SAMPLE ID (EPISODE #)	CLIENT CASE	DATE RECEIVED CLIENT SAMPLE ID	MATRIX/LEVEL	SAMPLE pH	% MOISTURE %	DRY WEIGHT g	AMOUNT EXTRACTED g (ml)	EXTRACTED VOLUME ml	GPC VOLUME ml	GPC ELUTE VOL ml	FLORISIL VOL CONC ml	FLORISIL FIN VOL CONC ml	VOL SV - P ml	FIN PROCESS VOLUME ml	SCREEN Y/N Y/N	MS SV - P µL	SURR SV - P µL	BASE pH CONFIRMATION	ACID pH CONFIRMATION	
BL1212WC			W/L				5.0	5.0					10.0	10.0	N		250			
BB1212WC			W/L				5.0	5.0					10.0	10.0	N		250			
LC1212WC			W/L				5.0	5.0					10.0	10.0	N	250	250			
LD1212WC			W/L				5.0	5.0					10.0	10.0	N	250	250			
45381.05 GC200	B&VSP-KC	12/08/2000 08:00 BV-BMC-RTN-002	W/L	6.22			5.0	5.0					10.0	10.0	N		250			A
45381.05MS GC200	B&VSP-KC	12/08/2000 08:00 BV-BMC-RTN-002	W/L	6.22			5.0	5.0					10.0	10.0	N	250	250			A
45381.05MSD GC200	B&VSP-KC	12/08/2000 08:00 BV-BMC-RTN-002	W/L	6.22			5.0	5.0					10.0	10.0	N	250	250			A

INITIALS

WEIGHING: JM	EXTRACTION: JM	FLORISIL:
CLEANUP:	KD:	Acetonitrile
BLOWDOWN:	TV:	
SURROG. SPIKER: JM	VERIFIER: GS	
MATRIX SPIKER: JM	VERIFIER: GS	
SUBMITTED BY: JM	REVIEWED BY: 12-13-00	

SOLVENTS

FLOBISIL CARTRIDGE #:
GPC COLUMN #:
SURROGATES ID CONC #: 6-155-8
MATRIX SPIKE SOLID/CONC #(s): 6-107-8
SOP - METHOD # : SWL-OP- 200 Rev. 5.0

Pg 30 BK1 Reog

COMMENTS

ATTACHMENT 1b
Sample Extraction Log — Explosives (GC200 — Soil)

[X] RUSH [ ] REEXTRACT [ ] TCLP [ ] DIOXIN

SAMPLE EXTRACTION LOG

	SV	ST • PCB	OTHER
CONT EX. (3520C)			
SEP-F (3510C)			
SHAKER			
SONIC. (3550B)			XXXXX
SOXHLET			

002423
Page: 1 of 2

BATCH: 010216SA

EXTRACT DATE: 2001-02-16 10:49

SAMPLE ID (EPISODE #)	CLIENT CASE	DATE RECEIVED CLIENT SAMPLE ID	MATRIX/LEVEL	SAMPLE pH	% MOISTURE %	DRY WEIGHT g	AMOUNT EXTRACTED g or ml	EXTRACTED VOLUME ml	GPC VOLUME ml	GPC ELUTE VOL ml	FLORISIL VOL CONC ml	FLORISIL FIN VOL CONC ml	VOL SV - P ml	FIN PROCESS VOLUME ml	SCREEN Y/N Y/N	MS SV - P µL	SURR SV - P µL	BASE pH CONFIRMATION	ACID pH CONFIRMATION	
BL0216SA			S/L				2.00	10.0						20.0	N		500			
BB0216SA			S/L				2.00	10.0						20.0	N		500			
LC0216SA			S/L				2.00	10.0						20.0	N	500	500			
LD0216SA			S/L				2.00	10.0						20.0	N	500	500			
45850.01 GC200	TOLTEST	02/16/2001 08:15 BI0520120031	S/L		30.78	1.6	2.26	10.0						20.0	N		500			A
45850.02MS GC200	TOLTEST	02/16/2001 08:15 BI0520120031	S/L		30.78	1.5	2.14	10.0						20.0	N	500	500			A
45850.03MSD GC200	TOLTEST	02/16/2001 08:15 BI0520120031	S/L		30.78	1.4	2.05	10.0						20.0	N	500	500			A
45850.04 GC200	TOLTEST	02/16/2001 08:15 BI0520120032	S/L		30.28	1.4	2.06	10.0						20.0	N		500			A
45850.05 GC200	TOLTEST	02/16/2001 08:15 BI0520120033	S/L		28.24	1.6	2.17	10.0						20.0	N		500			A
45850.06 GC200	TOLTEST	02/16/2001 08:15 BI0520120051	S/L		27.53	1.5	2.12	10.0						20.0	N		500			A
45850.07 GC200	TOLTEST	02/16/2001 08:15 BI0520120052	S/L		27.51	1.6	2.18	10.0						20.0	N		500			A
45850.08 GC200	TOLTEST	02/16/2001 08:15 BI0520120053	S/L		25.67	1.5	2.03	10.0						20.0	N		500			A
45850.09 GC200	TOLTEST	02/16/2001 08:15 BI0520120053FD	S/L		28.15	1.5	2.09	10.0						20.0	N		500			A
45850.10 GC200	TOLTEST	02/16/2001 08:15 BI0520120071	S/L		28.24	1.6	2.16	10.0						20.0	N		500			A
45850.11 GC200	TOLTEST	02/16/2001 08:15 BI0520120072	S/L		24.91	1.6	2.07	10.0						20.0	N		500			A

INITIALS

WEIGHING: GS	EXTRACTION: GS	FLORISIL:
CLEANUP:	KD:	Automatic/CaCl2
BLOWDOWN:	TV:	
SURROG. SPIKER: CD	VERIFIER: STR	
MATRIX SPIKER: CD	VERIFIER: STR	
SUBMITTED BY: CD	REVIEWED BY:	

2/17/01

ID NUMBERS

FLORISIL CARTRIDGE #:
GPC COLUMN #:
SURROGATES ID CONC #: 6-177-8
MATRIX SPIKE SOLID/CONC.#(s): 6-154-8
SOP - METHOD # : SWL-OP- 200 Rev. 4.1

Pg 32 Book 1 Reag

COMMENTS

ATTACHMENT 1b (Continued)
Sample Extraction Log — Explosives (GC200 — Soil)

[X] RUSH [ ] REEXTRACT [ ] TCLP [ ] OXIN

SAMPLE EXTRACTION LO

	SV	ST • PCB	OTHER
CONT EX. (3520C)			
SEP-F (3510C)			
SHAKER			
SONC. (3550B)			XXXXX
SOXHLET			

BATCH: 010216SA

EXTRACT DATE: 2001-02-16 10:49

002423
Page: 2 of 2

SAMPLE ID (EPISODE #)	CLIENT CASE	DATE RECEIVED CLIENT SAMPLE ID	MATRIX LEVEL	SAMPLE pH	% MOISTURE (%)	DRY WEIGHT (g)	AMOUNT EXTRACTED (g or ml)	EXTRACTED VOLUME (ml)	GPC VOLUME (ml)	GPC ELUTE VOL (ml)	FLORISIL VOL CONC (ml)	FLORISIL FIN VOL CONC (ml)	VOL SV - P (ml)	FIN PROCESS VOLUME (ml)	SCREEN Y/N (Y/N)	MS SV - P (µL)	SURR SV - P (µL)	BASE pH CONFIRMATION	ACID pH CONFIRMATION	
45850.12 GC200	TOLTEST	02/16/2001 08:15 BIOS20120073	S/L		25.94	1.5	2.02	10.0						20.0	N		500			A
45850.13 GC200	TOLTEST	02/16/2001 08:15 BIOS20120091	S/L		28.14	1.6	2.25	10.0						20.0	N		500			A
45850.14 GC200	TOLTEST	02/16/2001 08:15 BIOS20120092	S/L		25.34	1.5	2.03	10.0						20.0	N		500			A
45850.15 GC200	TOLTEST	02/16/2001 08:15 BIOS20120093	S/L		25.98	1.5	2.01	10.0						20.0	N		500			A
45850.16 GC200	TOLTEST	02/16/2001 08:15 BIOS20120111	S/L		28.76	1.5	2.13	10.0						20.0	N		500			A
45850.17 GC200	TOLTEST	02/16/2001 08:15 BIOS20120112	S/L		20.95	1.7	2.12	10.0						20.0	N		500			A
45850.18 GC200	TOLTEST	02/16/2001 08:15 BIOS20120113	S/L		25.74	1.5	2.07	10.0						20.0	N		500			A

INITIALS		
WEIGHING: GS	EXTRACTION: GS	FLORISIL:
CLEANUP:	KD:	Autoconcentrate/Cally
BLOWDOWN:	TV:	
SURROG. SPIKER: CD	VERIFIER: STR	
MATRIX SPIKER: CD	VERIFIER: STR	
SUBMITTED BY: CD	REVIEWED BY: 2/21/01	

ID NUMBERS	
FLORISIL CARTRIDGE #:	
GPC COLUMN #:	
SURROGATES/ID CONC #:	6-177-8
MATRIX SPIKE SOLID/CONC #(s):	6-154-8
SOP - METHOD # : SWL-OP-	200 Rev. 4.1

COMMENTS

Pg 32 Book 1 Reas

ATTACHMENT 1c
Sample Extraction Log — Explosives (GC201 — Soil)

(3520C)
SEP-F
(3510C)
SHAKER
SONC.
(3550B)
SOXHLET

XXXXX

002019
Page: 1 of 1

BATCH: 00110250

EXTRACT DATE: 2000-11-02 11:46

SAMPLE ID (EPISODE #)	CLIENT CASE	DATE RECEIVED CLIENT SAMPLE ID	MATRIX/LEVEL	SAMPLE pH	% MOISTURE %	DRY WEIGHT g	AMOUNT EXTRACTED (g) ml	EXTRACTED VOLUME ml	GPC VOLUME ml	GPC ELUTE VOL ml	FLORISIL VOL CONC ml	FLORISIL FIN VOL CONC ml	VOL SV - P ml	FIN PROCESS VOLUME ml	SCREEN Y/N Y/N	MS SV - P µL	SURR SV - P µL	BASE pH CONFIRMATION	ACID pH CONFIRMATION	Bot R
BL110250			S/L				2.02	10.0						20.0	N		500			
BB110250			S/L				2.00	10.0						20.0	N		500			
LC110250			S/L				2.01	10.0						20.0	N	500	500			
LD110250			S/L				2.00	10.0						20.0	N	500	500			
44877.01 GC201	TOLTEST	11/02/2000 08:15 MFBICS759	S/L		16.99	1.7	2.01	10.0						20.0	N		500			A
44877.01MS GC201	TOLTEST	11/02/2000 08:15 MFBICS759	S/L		16.99	1.7	2.00	10.0						20.0	N	500	500			A
44877.01MSD GC201	TOLTEST	11/02/2000 08:15 MFBICS759	S/L		16.99	1.7	2.03	10.0						20.0	N	500	500			A
44877.03 GC201	TOLTEST	11/02/2000 08:15 MFBICS761	S/L		17.44	1.7	2.01	10.0						20.0	N		500			A
44877.04 GC201	TOLTEST	11/02/2000 08:15 MFBICS762	S/L		19.43	1.6	2.04	10.0						20.0	N		500			A
44877.06 GC201	TOLTEST	11/02/2000 08:15 MFBICS764	S/L		20.68	1.6	2.01	10.0						20.0	N		500			A

INITIALS

WEIGHING:	GS	EXTRACTION:	GS	FLORISIL:	Acetonitrile CaCl2
CLEANUP:		KD:			
BLOWDOWN:		TV:			
SURROG. SPIKER:	GS	VERIFIER:	HM		
MATRIX SPIKER:	GS	VERIFIER:	HM		
SUBMITTED BY:	GS	REVIEWED BY:			11/2/2000

ID NUMBERS

FLORISIL CARTRIDGE #:
GPC COLUMN #:
SURROGATES ID CONC #: 6-145-8
MATRIX SPIKE SOLID/CONC #(s): 6-128-8
SOP - METHOD # : SWL-OP- 200 Rev. 5.0
Bo BZ Reagent

COMMENTS

ATTACHMENT 1d
Sample Extraction Log — Explosives (GC299 — Soil)

SHAKER			
SONC. (3550B)			XXXXX
SOXHLET			

EXTRACT DATE 12-11-13-04

002156
Page: 1 of 1

SAMPLE ID (EPISODE #)	CLIENT CASE	DATE RECEIVED CLIENT SAMPLE ID	MATRIX/LEVEL	SAMPLE pH	% MOISTURE %	DRY WEIGHT g	AMOUNT EXTRACTED g or ml	EXTRACTED VOLUME ml	GPC VOLUME ml	GPC ELUTE VOL ml	FLORISIL VOL CONC ml	FLORISIL FIN VOL CONC ml	VOL SV - P ml	FIN PROCESS VOLUME ml	SCREEN Y/N Y/N	µL	MS SV - P µL	SURR SV - P	BASE pH CONFIRMATION	ACID pH CONFIRMATION	Pg 4
BL1211SF			S/L				2.01	10.0						20.0	N		500				
BB1211SF			S/L				2.01	10.0						20.0	N		500				
LC1211SF			S/L				2.00	10.0						20.0	N	500	500				
LD1211SF			S/L				2.01	10.0						20.0	N	500	500				
45244.03 GC299	AVAL	12/01/2000 10:00 43797-03B	S/L		13.83	1.7	2.03	10.0						20.0	N		500				A
45244.04 GC299	AVAL	12/01/2000 10:00 43797-04B	S/L		17.54	1.7	2.01	10.0						20.0	N		500				A

DETAILS			
WEIGHING: CD	EXTRACTION: GS	FLORISIL:	
CLEANUP:	KD:	Automatic Cell	
BLOWDOWN:	TV:		
SURROG. SPIKER: GS	VERIFIER: SJR		
MATRIX SPIKER: GS	VERIFIER: SJR		
SUBMITTED BY: GS	REVIEWED BY:		

IDENTIFIERS	
FLORISIL CARTRIDGE #:	
GPC COLUMN #:	
SURROGATES ID CONC #:	6-155-8
MATRIX SPIKE SOLID/CONC #(s):	6-168-8
SOP - METHOD # : SWL-OP-	200 Rev. 5.0

COMMENTS
matrix spike - PETN

Pg 4 Bri Reagent

ATTACHMENT 2
Sample Extraction Log — Low Level Water (GC218 — Soil)

☒ RUSH ☐ REEXTRACT ☐ TCLP ☐ DIOXIN

SAMPLE EXTRACTION L

	SV	ST # PCB	OTHER
CONT EX. (3520C)			
SEP-F (3510C)			
SHAKER			XXXXX
SONC. (3550B)			
SOXHLET			

BATCH: 010221WA

EXTRACT DATE 2001-02-21 07:57

002432
Page: 1 of 1

SAMPLE ID (EPISODE #)	CLIENT CASE	DATE RECEIVED / CLIENT SAMPLE ID	MATRIX/LEVEL	SAMPLE pH	% MOISTURE	DRY WEIGHT	AMOUNT EXTRACTED	EXTRACTED VOLUME	GPC VOLUME	GPC ELUTE VOL	FLORISIL VOL CONC	FLORISIL FIN VOL CONC	VOL SV - P	FIN PROCESS VOLUME	SCREEN Y/N	MS SV - P	SURR SV - P	BASE pH CONFIRMATION	ACID pH CONFIRMATION	
					%	g	g (ml)	ml	ml	ml	ml	ml	ml	ml	Y/N	µl	µl			
BL0221WA			W/L				770	160					10.0	10.0	N		250			
BB0221WA			W/L				770	160					10.0	10.0	N		250			
LC0221WA			W/L				770	160					10.0	10.0	N	250	250			
LD0221WA			W/L				770	160					10.0	10.0	N	250	250			
45861.01 GC218	TOLTEST	02/20/2001 08:10 RPW077	W/L	6.57			770	160					10.0	10.0	N		250			bottle A
45861.01MS GC218	TOLTEST	02/20/2001 08:10 RPW077	W/L	6.57			770	160					10.0	10.0	N	250	250			B
45861.01MSD GC218	TOLTEST	02/20/2001 08:10 RPW077	W/L	6.57			770	160					10.0	10.0	N	250	250			B
45868.01 GC218	TOLTEST	02/21/2001 08:10 RPW078	W/L	7.03			770	160					10.0	10.0	N		250			A

NOTES

WEIGHING: MC	EXTRACTION: MC	FLORISIL:
CLEANUP:	KD:	Acetonitrile
BLOWDOWN:	TV:	water
SURROG. SPIKER: GS	VERIFIER: MC	
MATRIX SPIKER: GS	VERIFIER: MC	
SUBMITTED BY: MC	REVIEWED BY: 3/21/01	

NUMBERS

FLORISIL CARTRIDGE #:
GPC COLUMN #:
SURROGATES ID CONC #: 6-177-8
MATRIX SPIKE SOLID/CONC #(s): 6-154-8
SOP - METHOD # : SWL-OP 209 Rev. 6.0

COMMENTS

* Extracts had emulsion, needed centrifuged.

1932 bath / magnet

Typist: LLR

ATTACHMENT 3
Sample Extraction Log — Nitroglycerin (GC202 — Soil)

	SV	PCB	OTHER
CONT EX. (3520C)			
SEP-F (3510C)			
SHAKER			
SONC. (3550B)			XXXXX
SOXHLET			

(X) RUSH ( ) REEXTRACT ( ) TCLP ( ) DXIN

SAMPLE EXTRACTION L

BATCH: 010227SE

EXTRACT DATE: 01-02-27 12:04

002459
Page: 1 of 2

SAMPLE ID (EPISODE #)	CLIENT / CASE	DATE RECEIVED / CLIENT SAMPLE ID	MATRIX/LEVEL	SAMPLE pH	% MOISTURE %	DRY WEIGHT g	AMOUNT EXTRACTED g or ml	EXTRACTED VOLUME ml	GPC VOLUME ml	GPC ELUTE VOL ml	FLORISIL VOL CONC ml	FLORISIL FIN VOL CONC ml	VOL SV - P ml	FIN PROCESS VOLUME ml	SCREEN Y/N	MS SV - P µL	SURR SV - P µL	BASE pH CONFIRMATION	ACID pH CONFIRMATION
BL0227SE			S/L				2.00	10.0						20.0	N		500		
GB0227SE			S/L				2.01	10.0						20.0	N		500		
LC0227SE			S/L				2.00	10.0						20.0	N	500	500		
LD0227SE			S/L				2.00	10.0						20.0	N	500	500		
45903.01 GC202	CH2M-VA	02/26/2001 08:20 ASX01-SS0001-R01	S/L	7.72	23.98	1.7	2.21	10.0						20.0	N		500		B
45903.02 GC202	CH2M-VA	02/26/2001 08:20 ASX02-SS0001-R01	S/L	7.92	11.61	1.9	2.11	10.0						20.0	N		500		B
45903.03 GC202	CH2M-VA	02/26/2001 08:20 ASX03-SS0001-R01	S/L	6.74	18.30	1.7	2.08	10.0						20.0	N		500		B
45903.04 GC202	CH2M-VA	02/26/2001 08:20 ASX04-SS0001-R01	S/L	8.31	18.93	1.7	2.14	10.0						20.0	N		500		B
45903.05 GC202	CH2M-VA	02/26/2001 08:20 ASX05-SS0001-R01	S/L	7.85	14.64	1.9	2.22	10.0						20.0	N		500		B
45903.06 GC202	CH2M-VA	02/26/2001 08:20 ASX06-SS0001-R01	S/L	6.18	20.41	1.8	2.20	10.0						20.0	N		500		B
45903.07 GC202	CH2M-VA	02/26/2001 08:20 ASX08-SS0001-R01	S/L	7.46	20.95	1.7	2.17	10.0						20.0	N		500		B
45903.08 GC202	CH2M-VA	02/26/2001 08:20 ASX20-SS0001-R01	S/L	7.78	20.51	1.8	2.25	10.0						20.0	N		500		B
45903.09 GC202	CH2M-VA	02/26/2001 08:20 ASX07-SS0001-R01	S/L	7.93	13.82	2.0	2.30	10.0						20.0	N		500		B
45903.10 GC202	CH2M-VA	02/26/2001 08:20 ASX12-SS0001-R01	S/L	7.10	19.12	1.9	2.34	10.0						20.0	N		500		B
45903.11 GC202	CH2M-VA	02/26/2001 08:20 ASX19-SS0001-R01	S/L	8.10	19.23	1.9	2.35	10.0						20.0	N		500		B

INITIALS

WEIGHING:	EXTRACTION: GS	FLORISIL: Acetonitrile CH2Cl2	
CLEANUP:	KD:		
BLOWDOWN:	TV:		
SURROG. SPIKER: CD	VERIFIER: JF		
MATRIX SPIKER: CD	VERIFIER: JF		
SUBMITTED BY: CD	REVIEWED BY: [signature]		

ID NUMBERS

FLORISIL CARTRIDGE #:
GPC COLUMN #:
SURROGATES ID CONC #: 5:179:8
MATRIX SPIKE SOLID/CONC #(s): 5:178-8
SOP - METHOD # : SWL-OP-200 Rev. 5.0

COMMENTS

Pg 32 BK1 Rlog

ATTACHMENT 3 (Continued)
Sample Extraction Log — Nitroglycerin (GC202 — Soil)

	SV	PCB	OTHER
CONT EX. (3520C)			
SEP-F (3510C)			
SHAKER			
SONC. (3550B)			XXXXX
SOXHLET			

X: RUSH REEXTRACT TCLP DXIN

SAMPLE EXTRACTION L

BATCH: 010227SE

EXTRACT DATE: 01-02-27 12:04

002459

Page: 2 of 2

SAMPLE ID (EPISODE #)	CLIENT / CASE	DATE RECEIVED / CLIENT SAMPLE ID	MATRIX/LEVEL	SAMPLE pH	% MOISTURE %	DRY WEIGHT g	AMOUNT EXTRACTED (g) or ml	EXTRACTED VOLUME ml	GPC VOLUME ml	GPC ELUTE VOL ml	FLORISIL VOL CONC ml	FLORISIL FIN VOL CONC ml	VOL SV - P ml	FIN PROCESS VOLUME ml	SCREEN Y/N Y/N	MS SV - P µL	SURR SV - P µL	BASE pH CONFIRMATION	ACID pH CONFIRMATION	
45903.12 GC202	CH2M-VA	02/26/2001 08:20 ASX10-SS0001-R01	S/L	7.89	17.84	1.9	2.26	10.0						20.0	N		500			B
45903.13MS GC202	CH2M-VA	02/26/2001 08:20 ASX10-SS0001-R01	S/L	7.89	21.21	1.8	2.33	10.0						20.0	N	500	500			B
45903.14MSD GC202	CH2M-VA	02/26/2001 08:20 ASX10-SS0001-R01	S/L	7.89	18.75	1.9	2.38	10.0						20.0	N	500	500			B
45903.15 GC202	CH2M-VA	02/26/2001 08:20 ASX09-SS0001-R01	S/L	6.77	17.14	2.0	2.41	10.0						20.0	N		500			B
45903.16 GC202	CH2M-VA	02/26/2001 08:20 ASX11-SS0001-R01	S/L	5.75	14.62	2.0	2.30	10.0						20.0	N		500			B
45903.17 GC202	CH2M-VA	02/26/2001 08:20 ASX13-SS0001-R01	S/L	7.74	21.26	1.9	2.45	10.0						20.0	N		500			B
45903.18 GC202	CH2M-VA	02/26/2001 08:20 ASX14-SS0001-R01	S/L	8.25	20.45	1.7	2.17	10.0						20.0	N		500			B
45903.19 GC202	CH2M-VA	02/26/2001 08:20 ASX15-SS0001-R01	S/L	7.92	17.46	1.9	2.28	10.0						20.0	N		500			B
45903.20 GC202	CH2M-VA	02/26/2001 08:20 ASX16-SS0001-R01	S/L	8.00	15.40	1.8	2.14	10.0						20.0	N		500			B
45903.21 GC202	CH2M-VA	02/26/2001 08:20 ASX18-SS0001-R01	S/L	7.95	18.88	1.9	2.35	10.0						20.0	N		500			B
45903.22 GC202	CH2M-VA	02/26/2001 08:20 ASX16-SS0001P-R01	S/L	7.91	15.58	1.9	2.21	10.0						20.0	N		500			B

INITIALS			ID NUMBERS	COMMENTS
WEIGHING:	EXTRACTION: GS	FLORISIL:	FLORISIL CARTRIDGE #:	
CLEANUP:	KD:	CH2Cl2 Acetonitrile	GPC COLUMN #:	
BLOWDOWN:	TV:		SURROGATES ID CONC #: S-179-8	
SURROG. SPIKER: CD	VERIFIER: JF		MATRIX SPIKE SOLID/CONC #(s): S-178-8	
MATRIX SPIKER: CD	VERIFIER: JF			
SUBMITTED BY: CD	REVIEWED BY: 2-28-01		SOP - METHOD # : SWL-OP- 202 Rev. 50	

Pg 32 BKC1 Reos

SOUTHWEST LABORATORY OF OKLAHOMA, INC. *and*
AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.

TM

Standard Operating Procedure

Extraction of Herbicides by GC

Document No.: SWL-OE-411

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APPROVALS	DATES
Procedure Prepared By	9/19/2000 Date
Program Manager or Section Supervisor	9-19-2000 Date
Laboratory Safety Officer	9/20/2000 Date
Laboratory QA/QC Officer	9/19/2000 Date

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EXTRACTION OF HERBICIDES BY GC

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1.0 SCOPE AND APPLICATION

1.1 This method is designed to determine the concentrations of various acid herbicides in extracts from solid and liquid matrices. The method is based on EPA SW-846 methods 8000B and 8151A for analysis, extractions, and cleanups (Method 3580A is utilized for waste dilution.) The method can be used to determine analyte concentrations of herbicides in the ppb range with the electron capture detector (ECD.) Test codes encompassed by this method include but are not limited to GC550, GC560, GC570, GC571, GC580, and GC599.

1.2 Tables 1 and 2 list routinely analyzed herbicides using this method:

Table 1
Herbicide Target Compounds List (Full List)

COMPOUND NAME	CAS
2,4-D	94-75-7
2,4,-DB	94-82-6
2,4,5-TP (Silvex)	93-72-1
2,4,5-T	93-76-5
Dalapon	75-99-0
Dicamba	1918-00-9
Dichloroprop	120-36-5
Dinoseb	88-85-7
MCPA	94-74-6
MCPP	93-65-2
4-Nitrophenol	100-02-1
Pentachlorophenol	87-86-5

Table 2
Herbicide Pesticide Target Compounds List (Extended List)

COMPOUND NAME	CAS
2,4-D	94-75-7
2,4,-DB	94-82-6
2,4,5-TP (Silvex)	93-72-1
2,4,5-T	93-76-5
Dalapon	75-99-0
Dicamba	1918-00-9
Dichloroprop	120-36-5
Dinoseb	88-85-7
MCPA	94-74-6
MCPP	93-65-2
4-Nitrophenol	100-02-1
Pentachlorophenol	87-86-5
Acifluorfen	50594-66-6
Bentazon	25057-89-6
Chloramben	133-90-4
DCPA Diacid (a)	2136-79-0

(a) DCPA monoacid and diacid metabolites included in method scope; DCPA diacid metabolite used for validation studies. DCPA is a dimethyl ester.

1.3 There are no deviations from the method in this procedure.

2.0 METHOD SUMMARY

2.1 Method 8151A provides extraction, derivation and gas chromatographic conditions for analysis of chlorinated acid herbicides in water, soil, and waste samples. An option for the hydrolysis of esters is also described.

2.1.1 Water samples are extracted with diethyl ether and then esterified with diazomethane. The derivatives are determined by gas chromatography with an electron capture detector (GC/ECD.) The results are reported as acid equivalents.

2.1.2 Soil and waste samples are extracted and esterified with diazomethane. The derivatives are determined by GC/ECD. The results are reported as acid equivalents.

2.1.3 If herbicide esters are to be determined using this method, hydrolysis conditions for the esters in water and soil extracts are described.

2.2 A method blank, laboratory control spike, and a matrix spike/matrix spike duplicate must be prepared for each batch of 20 samples or less per matrix.

3.0 HEALTH AND SAFETY

3.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method (see Standard Operating Procedure SWL-GA-111, "*Laboratory Safety Plan*".) A reference file of Material Safety Data Sheets (MSDS) is available to all personnel involved in the chemical analysis.

3.2 Caution should be taken when working with all sample extracts and calibration standards. Samples are of unknown origin and should be considered hazardous. Many of the spiking solutions used, along with chlorinated solvents are considered carcinogens. Concentrated acids and bases, if not handled properly, can cause severe burns.

3.3 Gloves should be used for the manipulation of all sample extracts and calibration standards and preparation of all acid and base solutions. Wear personal protective equipment (PPE) and goggles or face shield when warranted. See the standard operating procedure SWL-GA-111, "*Laboratory Safety Plan*," for detailed safety procedures.

3.4 Diazomethane is a toxic carcinogen and can explode under certain conditions. The following precautions must be followed:

3.4.1 Use only a well-ventilated hood - do not breathe vapors.

3.4.2 Use a safety screen.

3.4.3 Use mechanical pipetting aides.

3.4.4 Do not heat above 90°C - EXPLOSION may result.

3.4.5 Avoid grinding surfaces, ground glass joints, sleeve bearings, glass stirrers - EXPLOSION may result.

3.4.6 Store away from alkali metals - EXPLOSION may result.

3.4.7 Solutions of diazomethane decompose rapidly in the presence of solid materials such as copper powder, calcium chloride, and boiling chips.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

4.1 Sample Extraction

4.1.1 Aqueous samples must be extracted within 7 days of collection. Solid samples must be extracted within 14 days of collection.

4.1.2 Extracts must be stored under refrigeration at 4°C (±2°C) and analyzed within 40 days of extraction.

4.2 Sample Storage

4.2.1 The samples must be protected from light and refrigerated at 4°C (±2°C) from the time of receipt until 60 days after delivery of a complete, reconciled sample data package to the client. After 60 days, the samples may be disposed of in a manner that complies with all applicable regulations.

4.2.2 The sample must be stored in an atmosphere demonstrated to be free of all potential contaminants.

4.2.3 Samples, sample extracts, and standards must be stored separately.

4.3 Sample Extract Storage

4.3.1 Sample extracts must be protected from light and stored at 4°C (±2°C).

4.3.2 Sample extracts must be stored in an atmosphere demonstrated to be free of all potential contaminants.

4.3.3 Samples, sample extracts, and standards must be stored separately.

4.4 All containers should be glass jars or bottles with Teflon lids. A minimum of one liter is required for aqueous samples (two liters is recommended). A minimum of 80g is required for soil samples, which includes enough additional sample for pH and percent moisture determination (125mL glass jar). One-hundred milliliters of TCLP extract is required for TCLP analysis.

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

5.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts or elevated baselines in gas chromatograms. All these materials must be analyzed routinely as reagent blanks to demonstrate the absence of interferences under the conditions of the analysis.

5.1.1 Glassware must be scrupulously cleaned. Clean each piece of glassware as soon as possible after use by rinsing it with the last solvent used. This should be followed by detergent washing with hot water and rinses with tap water, then with organic-free reagent water. Glassware should be solvent-rinsed with acetone and pesticide-quality hexane. After rinsing and drying, glassware should be sealed and stored in a clean environment to prevent any accumulation of dust or other contaminants. Store glassware inverted or capped with aluminum foil. Immediately prior to use, glassware should be rinsed with the next solvent to be used.

5.1.2 The use of high purity reagents and solvents helps to minimize interference problems. Purification of solvents by distillation in all-glass systems may be required.

5.2 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from sample to sample, depending upon the nature and diversity of the waste being sampled.

5.3 Organic acids, especially chlorinated acids, cause the most direct interference with the determination by methylation. Phenols, including chlorophenols, may also interfere with this procedure.

5.4 Alkaline hydrolysis and subsequent extraction of the basic solution removes many chlorinated hydrocarbons and phthalate esters that might otherwise interfere with the electron capture analysis. <u>However, hydrolysis may result in the loss of dinoseb and formation of aldol condensation products if any residual acetone remains from the extraction of soils</u>.

5.5 The herbicides, being strong organic acids, react readily with alkaline substances and may be lost during analysis. Therefore, glassware must be acid-rinsed and then rinsed to constant pH with organic-free reagent water. Sodium sulfate must be acidified.

5.6 Sample extracts must be dry prior to methylation or else poor recoveries will be obtained.

6.0 EQUIPMENT/APPARATUS

6.1 Separatory funnel - 2 liter, with Teflon stopcock.

6.2 Glass funnel with sodium sulfate.

6.3 Apparatus for grinding dry waste samples.

6.4 Apparatus for determining percent dry weight:

6.4.1 Oven - Drying.

6.4.2 Desiccator.

6.4.3 Crucibles - Porcelain or disposable aluminum.

6.5 Ultrasonic preparation — A horn type device equipped with a titanium tip, or a device that will give equivalent performance, shall be used.

Ultrasonic Disrupter — The disrupter must have a minimum power wattage of 300 watts, with pulsing capability. A device designed to reduce the cavitation sound is recommended. Follow the manufacturer's instructions for preparing the disrupter for extraction of samples.

6.6 Sonabox or equivalent — Recommended with above disrupters for decreasing cavitation sound (Heat Systems - Ultrasonics, Inc., Model 432B or equivalent).

6.7 Pasteur glass pipettes — 1mL, disposable.

6.8 Beakers — 400mL.

6.9 Vacuum or pressure filtration apparatus.

6.9.1 Buchner funnel.

6.9.2 Filter paper - S&S #595 (size 18.5 cm) or equivalent.

6.10 Kuderna-Danish (K-D) apparatus.

6.10.1 Concentrator tube — 10 mL, graduated (Kontes K-570050-1025 or equivalent). A ground-glass stopper is used to prevent evaporation of extracts.

6.10.2 Evaporation flask — 500 mL (Kontes K-570001-500 or equivalent). Attach to concentrator tube with springs or clips.

6.10.3 Snyder column — Three ball macro (Kontes K-503000-0121 or equivalent).

6.10.4 Springs — ½ inch (Kontes K-662750 or equivalent).

6.11 Solvent vapor recovery system (Kontes K-545000-1006 or K547300-0000, Ace Glass 6614-30, or equivalent).

6.12 Diazomethane generator

Diazald kit — Recommended for the generation of diazomethane (Aldrich Chemical Co., Catalog No. 210,050-0, or equivalent.)

6.13 Boiling chips — Solvent extracted and oven baked, approximately 10/40 mesh (silicon carbide or equivalent.)

6.14 Water bath — Heated, with concentric ring cover, capable of temperature control (±5°C). The bath should be used in a hood.

6.15 Balance — Top loading, capable of accurately weighing to the nearest 0.01 g to 100g.

6.16 Vials — Glass, 10-mL capacity with Teflon lined screw caps or crimp tops.

6.17 Glass scintillation vials — 20 mL, with aluminum lined screw caps.

6.18 pH indicator paper — narrow range (pH range of 1-6 and 7-11).

6.19 Syringe — 1.0, 2.5, and 10.0 mL.

6.20 Graduated cylinder — 1 liter.

6.21 Spatula — Stainless steel or Teflon.

6.22 Funnel — 72 mm diameter.

6.23 Wrist shaker — Burrell Model 75 or equivalent

6.24 Drying column — 400 mm X 20 mm ID Pyrex chromatographic column with Pyrex glass wool at bottom and a PTFE stopcock.

NOTE: Fritted glass discs are difficult to decontaminate after highly contaminated extracts have been passed through. Columns without frits may be purchased. Use a small pad of Pyrex glass wool to retain the adsorbent. Prewash glass wool pad with 50mL of acetone followed by 50mL of elution solvent prior to packing the column with adsorbent.

6.25 Erlenmeyer flask – 250 mL and 500 mL

6.26 Automated 8-place Glascol shaker or equivalent.

6.27 Turbovap extract concentrator.

6.28 Test tube rack.

6.29 Pyrex glass wool, acid washed.

6.30 Volumetric flasks, Class A: - 10 mL to 1000 mL.

6.31 Centrifuge

Centrifuge bottle 500 mL, Pyrex 1260 or equivalent

7.0 REAGENTS

7.1 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. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 Organic-free reagent water. All references to water in this method refer to organic-free water, as defined in Chapter One.

7.3 Sodium hydroxide solution (0.1 N), NaOH. Dissolve 4 g NaOH in organic-free reagent water and dilute to 1.0 L.

7.4 Potassium hydroxide solution (37% aqueous solution (w/v)), KOH. Dissolve 37g potassium hydroxide pellets in organic-free reagent water and dilute to 100mL.

7.5 Phosphate buffer pH = 2.5 (0.1 M). Dissolve 12g sodium phosphate (NaH_2PO_4) in organic-free reagent water and dilute to 1.0L. Add phosphoric acid to adjust the pH to 2.5.

7.6 N-methyl-N-nitroso-p-toluenesulfonamide (Diazald). High purity, available from Aldrich Chemical Co. or equivalent.

7.7 Silicic acid, H_2SiO_5. 100 mesh powder, store at 130°C.

7.8 Potassium carbonate, K_2CO_3.

7.9 2,3,4,5,6-Pentafluorobenzyl bromide (PFBBr), $C_6F_5CH_2Br$. Pesticide quality or equivalent.

7.10 Sodium sulfate (granular, acidified, anhydrous), Na_2SO_4. Purify by heating at 400°C for 4 hours in a shallow tray, or by precleaning the sodium sulfate with methylene chloride. If the sodium sulfate is precleaned with methylene chloride, a method blank must be analyzed, demonstrating that there is no interference from the sodium sulfate. Acidify by slurrying 100g sodium sulfate with enough diethyl ether to just cover the solid; then add 0.1mL of concentrated sulfuric acid and mix thoroughly. Remove the ether under vacuum. Mix 1g of the resulting solid with 5mL of organic-free reagent water and measure the pH of the mixture. It must be below a pH of 4. Store the remaining solid at 130°C.

7.11 Diazomethane Generation (Diazald Kit Method):

7.11.1 See Section 3, Health and Safety, before proceeding.

7.11.2 Assemble glassware. Set 400-mL beaker on hot plate and heat to 65°C.

7.11.3 Prepare reagents while the water bath is heating.

7.11.3.1 Dissolve 5 grams of KOH in 8 mL of water in a scintillation vial.

7.11.3.2 Dissolve 5 grams diazald in 45mL of ether in a 125mL Erlenmeyer flask.

7.11.4 Fill the condenser and receiver cooling bowl with dry ice. Add acetone slowly until full.

NOTE: Can use liquid nitrogen if dry ice is not available.

7.11.5 Add 14 mL of 2-(2-ethoxyethoxy)ethanol and 8 mL of ether to the potassium hydroxide solution in the reaction vessel.

7.11.6 Place the separatory funnel over the reaction vessel and transfer the diazald solution to the funnel.

7.11.7 When the water bath reaches 60-70°C, quickly add a small amount of diazald to start the reaction. Adjust so the rate of distillation is approximately equal to the rate of addition.

7.11.8 When all of the diazald has been added, rinse the diazald flask with about 10 mL of ether. Add the ether to the separatory funnel and continue the distillation process, until the distillate starts flowing colorless.

7.11.9 Remove the apparatus from the water bath. Pour the diazomethane from the receiver into VOA vials and refrigerate until ready for use. Use or dispose of the diazomethane within 2 weeks of preparation. Unused diazomethane is allowed to react in the hood with silica gel or methanol until the solution is colorless.

8.0 PROCEDURE

8.1 Upon receiving a copy of the LIMS (Laboratory Information Management System) sample log, the samples are retrieved from storage and logged out for extraction by the technician according to the chain-of-custody procedure.

8.2 The technician now initiates the automated extraction logsheet (See Attachment 1 for Extraction Log example). The pertinent extraction information is keyed into the Extraction Laboratory Database which is updated as the extraction procedure progresses.

8.3 After the samples are extracted and are ready for departmental transfer, the database-generated extraction log is reviewed and submitted with the sample extracts to the appropriate laboratory for analysis.

8.4 Aqueous Sample Procedure: Aqueous samples are extracted by Separatory Funnel Extraction (Method 8151A):

8.4.1 Using a 1 liter graduated cylinder, measure 1 liter (nominal) of sample and quantitatively transfer it to the 2-L separatory funnel. If high concentrations are anticipated, a smaller volume may be used and then diluted with organic-free reagent water to 1 liter. Add 0.5 mL of the surrogate standard to all samples, spikes, and blanks and mix well. For the sample in each analytical batch selected for spiking, add 1.0 mL of the matrix spiking standard and mix well.

8.4.2 Add 250g of NaCl to the sample, seal, and shake to dissolve the salt.

8.4.3 Use this step only if herbicide esters, in addition to herbicide acids, are to be determined.

Add 7mL of 10 N NaOH to the sample. Seal and shake the sample. Check the pH of the sample with pH paper. If the sample does not have a pH greater than or equal to 12, adjust the pH by adding more 10 N NaOH. Let the sample sit at room temperature until the hydrolysis step is completed (usually 1 –2 hours), shaking the separatory funnel and contents periodically.

8.4.4 Add 60mL of methylene chloride to the sample bottle and rinse both the bottle and the graduated cylinder. Transfer the methylene chloride to the separatory funnel and extract the sample by vigorously shaking the funnel for 2 minutes, with periodic venting to release excess pressure. Allow the organic layer to separate from the water phase for a minimum of 10 minutes. If the emulsion interface between the layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, but may include stirring, filtration through glass wool, centrifugation, or other physical methods.

Discard the methylene chloride phase. Extract 2 additional times with fresh 60mL portions of methylene chloride. Combine extracts.

8.4.5 Add 7mL of cold (4°C) 1:1 sulfuric acid to the sample (or hydrolyzed sample), seal, and shake to mix. Check the pH of the sample with pH paper. If the sample does not have a pH less than or equal to 2, adjust the pH by adding more acid.

8.4.6 Add 120mL of diethyl ether to the sample. Seal and extract the sample by vigorously shaking the funnel for 2 minutes with periodic venting to release excess pressure. Allow the organic layer to separate from the water phase for a minimum of 10 minutes. If the emulsion interface between layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum techniques to complete the phase separation depends upon the sample, but may include stirring, filtration through glass wool, centrifugation, or other physical methods. Remove the aqueous phase to a 2-L Erlenmeyer flask and collect the ether phase in a 500mL Erlenmeyer flask containing approximately 10g of acidified anhydrous sodium sulfate. Periodically, vigorously shake the extract and drying agent.

8.4.7 Return the aqueous phase to the separatory funnel, add 60mL of diethyl ether to the sample, and repeat the extraction procedure a second time, combining the extracts in the 500mL Erlenmeyer flask. Perform a third extraction with 60mL diethyl ether in the same manner. Allow the extract to remain in contact with the sodium sulfate for approximately 2 hours.

NOTE: the drying step is very critical to ensure complete esterification. Any moisture remaining in the ether will result in low herbicide recoveries. The amount of sodium sulfate is adequate if some free flowing crystals are visible when swirling the flask. If all of the sodium sulfate solidifies in a cake, add a few additional grams of acidified sodium sulfate and again test by swirling. The 2-hour drying time is a minimum, however, the extracts may be held in contact with the sodium sulfate overnight.

8.4.8 Pour the dried extract through a funnel plugged with acid washed glass wool, and collect the extract in the K-D concentrator. Use a glass rod to crush any caked sodium sulfate during the transfer

8.4.9 Assemble a Kuderna-Danish (K-D) concentrator by attaching a 10mL concentrator tube to a 500mL evaporation flask.

8.4.10 Add one or two clean boiling chips to the flask and attach a three-ball Snyder column. Prewet the Snyder column by adding about 1mL of diethyl ether to the top of the column. Place the K-D apparatus on a hot water bath (60-65°C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 45-60 minutes. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume of liquid reaches 1mL, allow to drain and cool for approximately 10 minutes.

8.4.11 Rinse the Snyder column into the sample extract with 1-2mL of diethyl ether, and transfer the extract to a scintillation vial.

8.4.12 Submit extracts to the GC Laboratory for derivation.

8.5 Soil Sample Procedure: Soil samples are extracted by the shaker method.

8.5.1 Sample Handling:

8.5.1.1 Sediment/soil samples — Decant and discard any water layer on a sediment sample. Mix sample thoroughly, especially composite samples. Discard any foreign objects such as sticks, leaves, and rocks.

8.5.1.2 Determine the dry weight of the sample remaining after decanting. Measurement of soil pH is required.

8.5.1.3 Waste samples — Samples consisting of multi-phases must be prepared by the phase separation method in Chapter Two of SW-846 before extraction. This procedure is for solids only.

8.5.1.4 Dry waste samples amenable to grinding — Grind or otherwise subdivide the waste.

8.5.1.5 Gummy, fibrous or oily materials not amenable to grinding should be cut, shredded, or otherwise broken up to allow mixing, and maximum exposure of the sample surfaces for extraction. The professional judgment of the analyst is required for handling of these difficult matrices.

8.5.1.6 Determination of percent moisture — weight out a portion of sample for this determination at the same time as the portion used for analytical determination.

WARNING: The drying oven should be contained in a hood or vented. Significant laboratory contamination may result from drying a heavily contaminated hazardous waste sample. However, samples known or suspected to contain significant concentrations of toxic, flammable, or explosive constituents should not be over-dried because of concerns for personal safety. Laboratory discretion is advised. It may be prudent to delay oven-drying of the weighed-out portion until other analytical results are available.

8.5.2 Shaker Extraction (Method 8151A):

8.5.2.1 Immediately after weighing the sample for extraction, weigh 5-10 g of the sample into a disposable aluminum weighing boat. Determine the % moisture of the sample by drying at 103°C (±2°C) for a predetermined amount of time or over night. Allow to cool before weighing. See Section 9.0, "Calculations" for calculation of percent moisture.

8.5.2.2 Add 50.0g of the well mixed, moist solid sample to a 500mL wide-mouth Erlenmeyer flask. Adjust the pH to 2 with concentrated HCl and monitor the pH for 15 minutes with occasional stirring. If

necessary, add additional HCl until the pH remains at 2. Add 0.5mL of the surrogate standard spiking solution onto the sample. For the sample in each analytical batch selected for spiking, add 1.0mL of the matrix spiking standard.

8.5.2.3 Add 20mL of acetone to the flask and mix the contents with a wrist shaker for 20 minutes. Add 80 mL of diethyl ether to the same flask and shake again for 20 minutes. Decant the extract and measure the volume of the solvent recovered.

8.5.2.4 Extract the sample twice more using 20mL of acetone followed by 80mL of diethyl ether. After addition of each solvent, the mixture should be shaken with a wrist shaker for 10 minutes and the acetone-ether extract decanted.

8.5.2.5 After the third extraction, the volume of extract recovered should be at least 75% of the volume of added solvent. If this is not the case, additional extractions may be necessary. Combine the extracts in a 2-L separatory funnel containing 250mL of reagent water. If an emulsion forms, slowly add 5g of acidified sodium sulfate (anhydrous) until the solvent-water mixture separates. A quantity of acidified sodium sulfate equal to the weight of the sample may be added, if necessary.

8.5.2.6 Check the pH of the extract. If it is not at or below pH 2, add more concentrated HCl until stabilized at the desired pH. Gently mix the contents of the separatory funnel for 1 minute and allow the layers to separate. Collect the aqueous phase in a clean beaker and the extract phase (top layer) in a 500mL ground glass-stoppered Erlenmeyer flask. Place the aqueous phase back into the separatory funnel and re-extract using 25mL of diethyl ether. Allow the layers to separate and discard the aqueous layer. Combine the ether extracts in a 500mL K-D flask.

8.5.2.7 <u>Use this step only if herbicide esters, in addition to herbicide acids, are to be determined</u>. If hydrolysis or additional cleanup is not required and the sample is dry, proceed to Section 8.5.3.3. Otherwise, proceed to Section 8.5.2.7.1 for hydrolysis or Section 8.5.3 for extract cleanup.

8.5.2.7.1 Add 5mL of 37% aqueous potassium hydroxide and 30mL of water to the extract. Add additional boiling chips to the K-D flask. Reflux the mixture on a water bath at 60-65°C until the hydrolysis step is completed (usually 1-2 hours). Remove the flasks from the water bath and cool to room temperature.

CAUTION: The presence of residual acetone will result in the formation of aldol condensation products, which will cause GC interference.

8.5.2.7.2 Transfer the hydrolized aqueous solution to a 500mL separatory funnel and extract the solution three times with 100mL portions of methylene chloride. Discard the methylene chloride phase. At this point, the basic (aqueous) solution contains the herbicide salts.

8.5.2.7.3 Adjust the pH of the solution to <2 with cold (4°C) sulfuric acid (1:3) and extract once with 40mL of diethyl ether and twice with 20mL portions of ether. Combine the extracts and pour them through a pre-rinsed drying column containing 7 to 10cm of acidified anhydrous sodium sulfate. Collect the dried extracts in a 500mL Erlenmeyer flask (with a 24/40 joint) containing 10g of acidified anhydrous sodium sulfate. Periodically, vigorously shake the extract and drying agent and allow the drying agent to remain in contact with the extract for a minimum of 2 hours.

NOTE: the drying step is very critical to ensure complete esterification. Any moisture remaining in the ether will result in low herbicide recoveries. The amount of sodium sulfate is adequate if some free flowing crystals are visible when swirling the flask. If all of the sodium sulfate solidifies in a cake, add a few additional grams of acidified sodium sulfate and again test by swirling. The 2-hour drying time is a minimum, however, the extracts may be held in contact with the sodium sulfate overnight.

8.5.2.7.4 Quantitatively transfer the contents of the flask to a 500mL Kuderna-Danish flask with a 10mL concentrator tube attached when the extract is know to be dry.

8.5.2.7.5 Proceed to Section 8.5.3.3 for extract concentration. If additional cleanup is required, proceed to Section 8.5.3.

8.5.3 Cleanup of non-hydrolized herbicide extracts:

8.5.3.1 Partition the herbicides by extracting the diethyl ether (8.5.2.7.4) three times with 15-mL portions of aqueous base prepared by carefully mixing 30 mL of reagent water into 15 mL of 37% aqueous potassium hydroxide. Discard the ether phase. At this point the basic (aqueous) solution contains the herbicide salts.

8.5.3.2 Adjust the pH of the solution to < 2 with cold (4°C) sulfuric acid (1:3) and extract once with 40 mL of diethyl ether and twice with 20-mL portions of ether. Combine the extracts and pour them through a pre-rinsed drying column containing 7-10 cm of acidified anhydrous sodium sulfate. Collect the dried extracts in a 500-mL Erlenmeyer flask (with a 24/40 joint) containing 10 g of acidified anhydrous sodium sulfate. Periodically, vigorously shake the extract and drying

agent and allow the drying agent to remain in contact with the extract for a minimum of 2 hours.

NOTE: the drying step is very critical to ensure complete esterification. Any moisture remaining in the ether will result in low herbicide recoveries. The amount of sodium sulfate is adequate if some free flowing crystals are visible when swirling the flask. If all of the sodium sulfate solidifies in a cake, add a few additional grams of acidified sodium sulfate and again test by swirling. The 2-hour drying time is a minimum, however, the extracts may be held in contact with the sodium sulfate overnight.

8.5.3.3 Quantitatively transfer the contents of the flask to a 500mL K-D flask with a 10mL concentrator tube attached when the extract is now to be dry.

8.5.3.4 Assemble a Kuderna-Danish (K-D) concentrator by attaching a 10mL concentrator tube to a 500mL evaporation flask.

8.5.3.5 Add one or two clean boiling chips to the flask and attach a three-ball Snyder column. Prewet the Snyder column by adding about 1mL of diethyl ether to the top of the column.

Place the K-D apparatus on a hot water bath (60-65°C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 45-60 minutes. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume of liquid reaches 1 mL, allow to drain and cool for approximately 10 minutes.

8.5.3.6 Rinse the Snyder column into the sample extract with 1-2mL of diethyl ether, and transfer the extract to a scintillation vial. The extracts are now submitted to the GC Laboratory for derivation (Section 8.5.3.7).

8.5.3.7 Reduce the extract volume to 2.5mL using nitrogen blowdown.

Place the scintillation vial under the apparatus and evaporate the solvent volume to a final volume of 2.5mL using a gentle stream of clean, dry nitrogen (filtered through a column of activated carbon).

CAUTION: Plastic tubing must not be used between the carbon trap and the sample, since it may introduce interferences.

8.5.3.8 Dilute the extract with 1mL of isooctane and 0.5mL of methanol. Dilute to a final volume of 4mL (if required) with diethyl ether.

8.5.3.9 Diazald kit method: Add 2mL of diazomethane solution and let the sample stand for 10 minutes with occasional swirling. The yellow color of diazomethane should be evident and should persist for this period.

8.5.3.10 Rinse the inside wall of the ampule with 700µL of diethyl ether. Reduce the extract volume to approximately 2mL (or until the extract turns clear) to remove excess diazomethane using an extremely gentle stream of clean, dry nitrogen. No condensation should be visible on the outside of the vial.

8.5.3.11 Dilute the sample to 10.0mL with hexane. Analyze by gas chromatography.

8.5.4 Waste Dilution (Method 3580A):

8.5.4.1 This method describes a solvent dilution of a non-aqueous waste sample prior to cleanup and/or analysis. It is designed for wastes that may contain organic chemicals at a concentration greater than 20,000 mg/Kg and that are soluble in the dilution solvent.

8.5.4.2 Samples consisting of multi-phases must be prepared by the phase separation method before extraction.

8.5.4.3 The sample dilution may be performed in a 10-mL volumetric flask. If disposable glassware is preferred, the 20-mL scintillation vial may be calibrated for use. Pipette 10.0 mL of extraction solvent (diethyl ether) into the scintillation vial and mark the bottom of the meniscus. Discard this solvent.

8.5.4.4 Transfer approximately 1g of each phase of the sample to separate 20mL vials or 10mL volumetric flasks (record weight to the nearest 0.1g). Wipe the mouth of the vial with a tissue to remove any sample material. Cap the vial before proceeding with the next sample to avoid any cross-contamination.

8.5.4.5 Add 1.0mL surrogate spiking solution to all samples and blanks. For the sample in each analytical batch selected for spiking, add 2.0mL of the matrix spiking standard.

8.5.4.6 Immediately dilute to 10mL with the diethyl ether.

8.5.4.7 Add 2.0g of anhydrous sodium sulfate to the sample.

8.5.4.8 Cap and shake the sample for 2 minutes.

NOTE: the drying step is very critical to ensure complete esterification. Any moisture remaining in the ether will result in low herbicide recoveries. The amount of sodium sulfate is adequate if some free flowing crystals are visible when swirling the flask. If all of the sodium sulfate solidifies in a cake, add a few additional grams of acidified sodium sulfate and again test by swirling. The 2-hour drying time is a minimum, however, the extracts may be held in contact with the sodium sulfate overnight.

8.5.4.9 Loosely pack disposable Pasteur pipettes with 2-3cm glass wool plugs. Filter the extract through the glass wool and collect 5mL of the extract in a tube or vial.

8.5.4.10 Submit extracts to the GC Laboratory for derivation. The GC Analyst will proceed to 8.5.3.7 for extract concentration and derivation.

8.5.4.11 Any reagent blanks and matrix spike samples must be subjected to exactly the same analytical procedures as those used on actual samples.

8.5.5 TCLP extraction:

8.5.5.1 Transfer 100mL of TCLP extract into a 250mL separatory funnel. Spike with 0.5mL of surrogate spike. Add 3mL of 1:1 sulfuric acid and check that the pH stabilizes at 2 or below. Addition of more sulfuric acid is required until stability at the desired pH is achieved.

8.5.5.2 Add 50mL of diethyl ether and shake for 1 minute. Drain the aqueous phase (bottom layer) into a beaker. Collect the organic phase (top layer) into a boiling flask containing 2mL of 37% KOH and 15 mL of deionized water. Add several boiling chips.

8.5.5.3 Repeat two additional times using 50mL of diethyl ether each time.

8.5.5.4 Place the boiling flask with combined organic phase into a water bath at 60-65°C for 1 hour.

8.5.5.5 Remove the boiling flask from the bath and let cool. Remove the Snyder column and transfer back into a separatory funnel. Rinse the flask twice with approximately 5mL of deionized water each time.

8.5.5.6 Check that the pH is basic.

8.5.5.7 Add 20mL of diethyl ether to the separatory funnel and shake for 1 minute. Collect the aqueous phase (bottom layer) in a beaker. Discard organic phase (top layer).

8.5.5.8 Repeat once more with 20mL of diethyl ether to wash the basic solution.

8.5.5.9 Add 2mL of 1:1 sulfuric acid to the aqueous phase. The pH must stabilize a 2 or below.

8.5.5.10 Add 20mL of diethyl ether to the separatory funnel and shake for 1 minute. Drain the bottom layer into a beaker. Collect the top layer into an Erlenmeyer flask containing 2-4 grams of acidified anhydrous sodium sulfate.

8.5.5.11 Repeat the process with 20mL of ether two additional times.

8.5.5.12 Funnel the extract with filter paper and approximately 10 grams of acidified anhydrous sodium sulfate. Rinse well with diethyl ether.

8.5.5.13 Assemble a K-D concentrator by attaching a 10mL concentrator tube to a 500mL evaporation flask.

8.5.5.14 Add one or two clean boiling chips to the flask and attach a three-ball Snyder column. Prewet the Snyder column by adding about 1mL of diethyl ether to the top of the column. Place the K-D apparatus on a hot water bath (60-65°C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 45-60 minutes. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume of liquid reaches 1mL, allow to drain and cool for approximately 10 minutes.

8.5.5.15 Rinse the Snyder column into the sample extract with 1-2mL of diethyl ether, and transfer the extract to a scintillation vial. The extracts are now submitted to the GC Laboratory for derivation (Section 8.5.3.7).

9.0 CALCULATIONS

Dry weight calculation (from data gathered during step 8.5.1.2):

$$\% \text{ Moisture} = \frac{\text{grams of sample - grams of dried sample}}{\text{grams of sample}} \times 100$$

10.0 QUALITY ASSURANCE/QUALITY CONTROL

Every 20 samples or sample delivery group (SDG) is considered a batch. Two method blanks, laboratory control spikes, and a matrix spike (MS) and matrix spike duplicate (MSD) are extracted with each SDG of up to 20 samples. In most cases, a client will specify a sample for MS/MSD. If MS/MSD sample is not specified, the laboratory chooses one at their discretion. Also, if two or more clients send samples not totaling 20 in number, one set of method blanks, LCS and MS/MSD are applied to both clients' samples.

11.0 WASTE DISPOSAL/POLLUTION PREVENTION

See the Standard Operating Procedure SWL-GA-114, "*Hazardous Waste Management Plan.*"

12.0 METHOD PERFORMANCE

The MDL concentrations are updated annually and are obtained using organic-free reagent water and sandy loam soil. The MDL results are maintained on file in the laboratory. Details for determining MDLs are given in the SOP "*Definition and Determination of Detection Limits*", SWL-GA-113. The MDL actually achievable in a given analysis will vary depending on detector response characteristics, irreducible noise from instrument electronics and matrix effects.

13.0 REFERENCES

U.S.EPA SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods: "Method 3500B. Organic Extraction and Sample Preparation" Revision 2, December 1996.

U.S.EPA SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods: "Method 3580A. Waste Dilution" Revision 1, July 1992.

U.S.EPA SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods: "Method 8000B. Determinative Chromatographic Separations" Revision 2, December 1996.

U.S.EPA SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods: "Method 8151A. Chlorinated Herbicides by GC Using Methylation or Pentafluorobenzylation Derivatization" Revision 1. December 1996.

Standard Operating Procedure, "*Laboratory Safety Plan*", SWL-GA-111.

Standard Operating Procedure, "*Control Charts*", SWL-GA-112.

Standard Operating Procedure, "*Hazardous Waste Management Plan*", SWL-GA-114.

14.0 DEFINITIONS

PQL:..... The Practical Quantitation Limit.

N/A:..... Not applicable

Material Safety Data Sheet: .... (MSDS) Information provided by commercial vendors for the chemical products prepared by their company. The information on the MSDS includes but is not limited to composition, physical properties, hazards identification, first aid measures, emergency clean-up and/or response, exposure, and storage.

SDG: ..... The Sample Delivery Group) A unit within a single case that is used to identify a group of samples for delivery. A SDG is a group of 20 or fewer field samples within a case, received over a period of up to 14 calendar days (7 calendar days for 14-day turns). Data from all samples in a SDG are due concurrently.

15.0 ATTACHMENTS

15.1 Attachment 1: Sample Extraction Log

ATTACHMENT 1
Sample Extraction Log

CONT EX. (3520C)			
SEP-F (3510C)		XXXXX	
SHAKER			
SONC. (3550B)			
SOXHLET			

BATCH: 000

EXTRACT DATE 2000-08-31 12:38

001748

Page: 1 of 1

SAMPLE ID (EPISODE #)	CLIENT CASE	DATE RECEIVED CLIENT SAMPLE ID	MATRIX/LEVEL	SAMPLE pH	% MOISTURE %	DRY WEIGHT g	AMOUNT EXTRACTED g or ml	EXTRACTED VOLUME ml	GPC VOLUME ml	GPC ELUTE VOL ml	FLORISIL VOL CONC ml	FLORISIL FIN VOL CONC ml	VOL SV (P) ml	FIN PROCESS VOLUME ml	SCREEN Y/N Y/N	MS SV (P) µL	SURR SV (P) µL	BASE pH CONFIRMATION	ACID pH CONFIRMATION	Bottle
BL0831WC			W/L				1000	300					10.0	10.0	N		1000			
BB0831WC			W/L				1000	300					10.0	10.0	N		1000			
LC0831WC			W/L				1000	300					10.0	10.0	N	1000	1000			
LD0831WC			W/L				1000	300					10.0	10.0	N	1000	1000			
44130.04 GC601	BECJACOB	08/29/2000 10:00 SA22	W/L	7.11			1000	300					10.0	10.0	N		1000			A
44130.05MS GC601	BECJACOB	08/29/2000 10:00 SA22	W/L	7.11			1000	300					10.0	10.0	N	1000	1000			AB
44130.06MSD GC601	BECJACOB	08/29/2000 10:00 SA22	W/L	7.10			1000	300					10.0	10.0	N	1000	1000			AB
44130.17 GC601	BECJACOB	08/29/2000 10:00 SA36	W/L	8.01			1000	300					10.0	10.0	N		1000			A
44130.22 GC601	BECJACOB	08/29/2000 10:00 SA92	W/L	6.92			1000	300					10.0	10.0	N		1000			A
44130.27 GC601	BECJACOB	08/29/2000 10:00 SA175	W/L	7.38			1000	300					10.0	10.0	N		1000			A
44130.32 GC601	BECJACOB	08/29/2000 10:00 SA8	W/L	7.10			1000	300					10.0	10.0	N		1000			A
44130.37 GC601	BECJACOB	08/29/2000 10:00 SA64	W/L	7.06			1000	300					10.0	10.0	N		1000			B
44151.01 GC601	B&WC	08/30/2000 09:50 LTANK1	W/L	7.48			1000	300					10.0	10.0	N		1000			A

INITIALS

WEIGHING:	SA	EXTRACTION:	JM	FLORISIL:	
CLEANUP:		KD:	CD		
BLOWDOWN:	JM	TV:			
SURROG. SPIKER:	JM	VERIFIER:	GS		
MATRIX SPIKER:	JM	VERIFIER:	GS		
SUBMITTED BY:	JM	REVIEWED BY:			

ID NUMBERS

FLORISIL CARTRIDGE #:

GPC COLUMN #:

SURROGATES ID CONC #: 5-218-5

MATRIX SPIKE SOLID/CONC #(s): 5-219-5

SOP - METHOD # : SWL-OP- 415 Rev. 3.0

COMMENTS

Acid washed

Pg 29 BK1 Rec8

SOUTHWEST LABORATORY OF OKLAHOMA, INC. *and*
AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.

TM

Standard Operating Procedure

Extraction of Semivolatile Organic BNA's by SW846 Methods: 3510C, 3520C, 3550B, and EPA Method 625

Document No.: SWL-OE-500

Rev No. / Date: Rev. 5.1 — 01/25/01

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APPROVALS	DATES
Procedure Prepared By	1/25/2001 Date
Program Manager or Section Supervisor	1-26-01 Date
Laboratory Safety Officer	1/29/2001 Date
Laboratory QA/QC Officer	1-27-01 Date

(Effective Date is 5 calendar days after the last signature above - QA/QC Officer)

Document Status

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EXTRACTION OF SEMIVOLATILE ORGANIC BNA's by
SW846 METHODS: 3510C, 3520C, 3550B, AND EPA METHOD 625

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1.0 SCOPE AND APPLICATION

The (SWL-OE-500) SOP is used for the extraction of semivolatile organic compounds in methylene chloride or a mixture of methylene chloride/acetone, for all types of matrices, such as: ground and waste waters, soils, oils, and all other types of solid waste matrices. This extraction procedure is used for the following Southwest Laboratory internal test codes. MS100, MS500, MS505, MS506, MS507, MS510, MS530, MS540, MS550, MS566 and when applicable MS599 (see special instruction for all MS599 codes.)

2.0 METHOD SUMMARY

2.1 This method is for the extraction of semivolatile (BNA) organic compounds and is based on SW846, Update III, Dec. 1997 methods 3510C (Separatory Funnel), 3520C (Continuous Liquid-Liquid) and 3550B (sonication), and has been written into a procedure for Southwest Laboratory of Oklahoma, Inc. For this SOP method 3510C will be noted as Separatory Funnel, Sep. Funnel, or SEP-F, method 3520C will be noted as Liquid-Liquid, continuous liquid-liquid, or continuous extractor (cont. ex.), and method 3550B will be noted as sonication or sonc.

2.2 A measured volume, usually 1 liter, of aqueous liquid is extracted with methylene chloride by either a separatory funnel or liquid - liquid extractor, prior to extraction the liquid must be adjusted to a pH < 2 and 2nd by adjusting sample to pH >11. This methylene chloride extract is dried, by use of a drying agent and concentrated to a final of 1mL.

2.3 A measured weight, (normally 30 grams for low level organics or 1 gram for medium/high level organics) of solid or non-aqueous liquid, is mixed with a drying agent and, extracted with a methylene chloride/acetone mixture by sonication. This extract is concentrated to a final volume of 1mL, or 0.5mL, if GPC work is done.

3.0 HEALTH AND SAFETY

3.1 Caution should be taken when extracting all samples. Most samples are of unknown origin and should be considered hazardous. Many of the spiking solutions used, along with chlorinated solvents are considered carcinogenic. Concentrated acids and bases, if not handled properly, can cause severe burns.

3.2 Gloves should be used for the extraction of all samples and preparation of all acid and base solutions. Wear personal protective equipment (PPE) and goggles or face shield when warranted. See SOP SWL-GA-111, "*Laboratory Safety Plan*" for detailed safety procedures.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

4.1 Extraction holding times for samples are as follows: 7 days from the date of collecting aqueous samples and 14 days from the date of sampling for soil/solid samples.

4.2 No sample preservation is necessary for organic semivolatiles, however samples and final extracts must be kept at 4 degrees Celsius ± 2 degrees.

4.3 All containers should be glass jars or bottles with Teflon lids. A minimum of one liter is required for aqueous samples. A minimum of 30 grams is required for soil samples, (125mL glass jar).

Note: Amber bottles should be used for aqueous samples.

5.0 INTERFERENCE'S AND POTENTIAL PROBLEMS

5.1 Extreme care must be taken when extracting samples in an effort to minimize laboratory contamination. This contamination can come from unclean glassware, gloves, and any other apparatus being used in the extraction area. The use of many types of protective equipment, especially gloves, contain high levels of phthalates. Many of these phthalates interfere with the analysis of target compounds. Therefore, extreme caution must be taken. One must refrain from touching the inside of any container, any solvent, or any apparatus when wearing gloves.

5.2 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or cause interference's to sample analysis. All these materials must be demonstrated to be free from interference under the conditions of the analysis by analyzing method blanks. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be required.

5.3 Phthalate ester contamination is commonly found in the laboratory. Plastics, in particular, must be avoided because phthalates are commonly used as plasticizers and are easily extracted from plastic materials. Serious phthalate contamination may result at any time if consistent quality control is not practiced.

5.4 Glassware contamination resulting in analyte degradation: Soap residue on glassware may cause degradation of certain analytes. Specifically, aldrin, heptachlor, and most organophosphorus pesticides will degrade in this situation. This problem is especially pronounced with glassware that may be difficult to rinse (e.g., 500mL K-D flask, synder columns, and condenser tubes). These items should be hand-rinsed very carefully to avoid this problem (See SOP SWL-OE-101, "*Organic Glassware*").

6.0 EQUIPMENT AND APPARATUS

6.1 General Extraction Equipment and Apparatus

6.1.1 400 mL Pyrex beakers

6.1.2 pH meter, (Orion 420A), or equivalent.

6.1.3 pH - paper, narrow range (1 - 6) and (7 -11).

6.1.4 Zymark (turbovap), nitrogen blowdown equipment, or equivalent.

6.1.5 Zymark (turobovap) extract blowdown tubes, (1 & ½ mL tips), or equivalent.

6.1.6 Short stem glass funnels: top ID 100 mm, stem 100 mm, (Pyrex or Kimax)

6.1.7 filter paper, (Schleicher & Schuell #595), or equivalent.

6.1.8 Kuderna Danish concentrator apparatus (500 ml)

6.1.9 3 ball snyder columns

6.1.10 10 mL concentrator tubes

6.1.11 (5 ¾ or 9) in. disposable pipettes

6.1.12 1.8 mL extract vials (wheaton or equivalent)

6.1.13 20 mL scintillation vials (wheaton or equivalent)

6.1.14 PFE-Latex Gloves, (Safeskin or equivalent)

6.1.15 temperature controlled hot water bath.

6.1.16 glass stirring rods

6.2 Soil Extraction Equipment and Apparatus

6.2.1 Stainless steel spatulas

6.2.2 Sonicators (Vibra Cell model VC602) or equivalent, with ¾ in. probe or 4-post micro probe.

6.2.3 GPC - Low Pressure - ABC 1002A or ABC 1002B - or equivalent.

6.2.4 GPC - High-Pressure - Zymark (Benchmate) dual column, Marathon single column, or equivalent.

6.2.5 Aluminum weighting Pans (70 X 16 mm).

6.2.6 Electronic Balance (OHAUS Galaxie 400 or equivalent).

6.2.7 10 mL PP+PE sterile disposable syringes

6.2.8 Acrodics, 0.45 micron, (Gelman or equivalent.)

6.2.9 Automated Shaker

6.3 Water Extraction Equipment and Apparatus

6.3.1 2000 mL Separatory Funnels

6.3.2 1000 mL Graduated cylinders

6.3.3 500 mL boiling flasks

6.3.4 Liquid - Liquid extractors capable or holding 1300 mL of liquid.

6.3.5 Boiling Beads (Hengar Granules plain and micro) or equivalent.

6.3.6 Automated electrical heating panel to control the 18-hr. extraction setting for liquid-liquid extractors.

6.3.7 Automated Separatory funnel shaker (8 - port).

6.3.8 8-port Separatory funnel and filter holder

6.3.9 4 oz. specimen containers (polypropylene with polypropylene caps).

7.0 REAGENTS AND SOLVENTS

7.1 Reagents

7.1.1 Reagent Water, ASTM type II, as described in the water quality SOP

7.1.2 Sulfuric Acid (Fisher certified A.C.S. PLUS) or equivalent.

7.1.3 Sodium Hydroxide pellets (Fisher certified A.C.S.) or equivalent.

7.1.4 Sodium Sulfate, Anhydrous crystals (JOST low organic A.C.S.) or equivalent.

7.2 Solvents

7.2.1 Methanol (Fisher A.C.S. grade spectra analyzed) or equivalent.

7.2.2 Methylene Chloride (Fisher OPTIMA), or equivalent.

7.2.3 Acetone (Fisher GC Resolv), or equivalent.

8.0 PROCEDURE

8.1 Extraction of Base/Neutral/Acid Fractions from Water

8.1.1 Retrieve water samples from sample storage, and fill out sample internal chain of custody records before removing samples from sample storage area.

8.1.2 Retrieve water extraction log book and fill out pertinent information: Client, Date received, Extraction Date, SWLO Sample ID, client sample ID, Extraction type, matrix, etc.

8.1.3 Prepare preprinted extraction labels for all samples and QC, each sample should have 4 preprinted labels to be used during extraction and final blowdown.

8.1.4 Make pH determination of each sample (see section 8.6.2), if pH is < 5 or > 9 pH units, inform the supervisor of the pH values on these samples. Record pH values on Extraction Log for each sample including Matrix Spike and Duplicate.

8.1.5 Proceed to section 8.2 for method 3510C (separatory funnel extraction) or section 8.3 for method 3520C (liquid-liquid extraction).

8.2 Method 3510C (Separatory Funnel Extraction)

8.2.1 Prerinse all glassware and apparatus used with extraction solvent.

8.2.2 Set up 2-liter separatory funnel in 8-port Sep. funnel holder (top Level)

8.2.3 Set up glass filter funnels, with S&S # 595 filter paper, and approximately 100 grams of Sodium Sulfate, in 8-port Sep. funnel holder (middle level). Record Sodium Sulfate lot # on Extraction Log, in comment section.

8.2.4 Set up Kuderna Danish concentrator apparatus with boiling chips in the 8-port Sep. funnel holder, if necessary a 500mL boiling flask with boiling chips may be substituted (lower level).

8.2.5 Install preprinted sample ID labels on Sep. funnel and kuderna Danish concentrator on boiling flask.

8.2.6 Retrieve the appropriate number of 1000mL Graduated cylinders, one for each sample + QC.

8.2.7 Shake sample bottle and pour sample into 1000mL Graduated cylinder. 1000mL of sample should be used. Record actual volume to nearest ½ graduated cylinder mark on the extraction log. Pour approximately 10-15ml of sample into a scintillation vial and take pH reading immediately and record in Extraction Log.

8.2.8 Pour sample from graduated cylinder into 2-liter Sep. funnel, care must be taken to make sure that correct sample is poured into correctly labeled Sep. funnel.

8.2.9 Continue doing 8.2.7 and 8.2.8 for each sample plus QC being extracted. For blanks and laboratory control spikes, Reagent water will be used in section 8.2.7.

Note: After all samples are poured out all remaining sample must be returned to the sample storage area and the internal chain of custody records must be filled to indicate that the remaining sample has been returned. If the entire sample was used for the extraction, then the pitched bottle Log must be filled out. This log is with the internal chain of custody records.

8.2.10 pH Adjustment — If samples are to have both Base/neutral and Acid or Base/neutral only compounds tested continue with 8.2.10.1. and skip section 8.2.10.2.

If samples are for Acid Extraction only (MS505 test code) then skip section 8.2.10.1 and start with section 8.2.10.2.

8.2.11 Surrogate Spike Addition

To each sample + QC add 500uL of surrogate spiking solution. Check to verify that you have the correct surrogate spiking solution, for the test code being

performed, and that the surrogate spiking solution is not expired. Record surrogate spiking solution ID #, the spiking of each sample, and sample spike verification (second person) on Extraction Log.

Note: The surrogate spiking solution is prepared and tested by the GC/MS Semivolatile analyses Dept. prior to use. When the solution is approximately 50 percent used up or expired, a new solution must be requested from the Semivolatile Dept.

8.2.12 Laboratory Control Spike (LCS) Addition

To each Laboratory Control Sample (LCS) or duplicate (LCSD) add 500uL of LCS spiking solution. Check to verify that you have the correct LCS spiking solution, for the test code being performed, and that the LCS spiking solution is not expired. Record LCS spiking solution ID #, the spiking of LCS samples, and sample spike verification (second person) on Extraction Log. This is normally the same solution as the Matrix Spike Solution and is recorded in the same location on the Extraction Log. When performing Method 625, addition of all client specified compounds of interest is required.

Note: The laboratory control spiking solution is prepared and tested by the GC/MS Semivolatile analyses Dept. prior to use. When the solution is approximately 50 percent used up or expired, a new solution must be requested from the Semivolatile Dept.

8.2.13 Matrix Spike (MS) Addition

To each Matrix Spike Sample (MS) or duplicate (MSD) add 500uL of MS spiking solution. Check to verify that you have the correct MS spiking solution, for the test code being performed, and that the MS spiking solution is not expired. Record the MS spiking solution ID #, the spiking of MS samples, and sample spike verification (second person) on Extraction Log. This is normally the same solution as the Laboratory Control Spike Solution. When performing Method 625, addition of all client specified compounds of interest is required. The matrix spike spiking solution is prepared and tested by the GC/MS Semivolatile analyses Dept. prior to use. When the solution is approximately 50% used up or expired, a new solution must be requested from the Semivolatile Dept.

8.2.14 Acid pH adjustment — To each sample + QC slowly add (1:1) sulfuric acid until a pH value less than 2 is constant by testing with narrow range pH paper (1 - 6). Sample should be stirred with a glass stirring rod, or stopper the Sep. funnel and hand shake for approximately 5 seconds, test the pH and again stir the sample and test the pH again to verify proper pH. Record pH change on Extraction Log under Acid pH confirmation and note pH change with <2.

8.2.15 Base/Neutral pH adjustment — To each sample + QC slowly add (10N sodium hydroxide) until a pH value greater than 11 is constant by testing with narrow range pH paper (7 - 11). Sample should be stirred with a glass stirring rod or stopper the Sep. funnel and hand shake for approximately 5 seconds, then test the pH and again stir the sample and check the pH again to verify proper pH. Record

pH change on Extraction Log under Base pH confirmation and note pH change with >11.

8.2.16 Extraction Solvent Addition — To each sample + QC add 60mL of methylene chloride. Record the methylene chloride Lot # on the Extraction Log, in the comments section.

8.2.17 Sample Extraction

8.2.17.1 Place the stopper in the Sep. funnel, hand - shake for approximately 10 seconds, and then vent in a ventilation hood. Repeat twice more the 10 second shaking and venting. (This process is to release any pressure build up cause by the extraction solvent).

8.2.17.2 Take Sep. funnel and install on 8 port automatic shaker and set timer for 3 minutes. When shaker stops, remove Sep. funnel immediately, vent and put back in the original place on 8-port Sep. funnel holder, (check sample IDs on Sep. funnels and KDs to ensure that samples do not get switched).

8.2.17.3 Allow Sep. funnel to set undisturbed for a minimum of 10 minutes to allow aqueous and solvent phases to separate.

Note: If an emulsion interface between the layers forms and cannot be broken by the use of physical means (i.e., stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods), the sample should be extracted in a continuous extractor or separated into more than one Sep. Funnel (to increase the solvent to sample amount), in an attempt to decrease the emulsion. Contact supervisor for instructions, before proceeding, if emulsion is problematic.

8.2.17.4 Slowly open stop - cock on Sep. funnel and let methylene chloride drain from Sep. funnel onto the sodium sulfate and into the KD.

Note: the sodium sulfate needs to be saturated with methylene chloride prior to drying extract. If this is not done it is very possible that part of the sample extract will be held up in the sodium sulfate.

8.2.17.5 After methylene chloride has drained out close stop - cock and repeat steps (8.2.14.1 - 8.2.15.4) two additional times.

8.2.17.6 If Base Extraction is also needed on the samples then proceed to section 8.1.15.9 other wise continue on to section 8.2.15.8.

8.2.17.7 Check pH of extracted water to verify that pH is still <2, if not then readjust pH and repeat steps (8.2.14.1 – 8.2.15.4) two additional times.

8.2.17.8 Check Sodium Sulfate: if the sodium sulfate has hardened, then the methylene chloride extract must be re-dried using new sodium sulfate.

This must be done before sample extract is concentrated. If only Base/neutral extract is being done (MS507) and the extract is deemed to be dry then proceed to section 8.2.17.

8.2.17.9 Base pH adjustment

To each sample + QC slowly add (10 N) Sodium Hydroxide until a pH value >11 is constant by testing with narrow range pH paper (7-11). Sample should be stirred with a glass stirring rod, or stopper the Sep. funnel and hand - shake for approximately 5 seconds, then test the pH and again stir the sample and check the pH again to verify proper pH. Record pH change on Extraction Log under Base pH confirmation and note pH change with >11.

8.2.17.10 After base pH is adjusted return to section 8.2.14 and continue procedure through section 8.2.15.5.

8.2.17.11 Check pH of extracted water to verify that pH is still >11, if not then readjust pH and repeat steps in sections (8.2.14.1 - 8.2.15.4) two additional times.

8.2.17.12 Check Sodium Sulfate, if the sodium sulfate has hardened then the methylene chloride extract must be re-dried using new sodium sulfate. This must be done before the sample extract is concentrated. When the extract is deemed to be dry then proceed to section 8.2.16.

8.2.18 Initial Extract Concentration

8.2.18.1 Attach a three-ball Snyder column to the KD. Pre-wet the column with approximately 1mL of methylene chloride.

8.2.18.2 Place the KD assembly in a hot water bath (not above 65°C) so that the entire lower rounded surface of the KD assembly is immersed in the water. The position of the KD assembly in the water bath and the water bath temperature should be such that the concentration is completed in 45-60 minutes. The balls of the Snyder column should actively chatter and the chambers should not flood with condensed solvent.

8.2.18.3 When the apparent volume of liquid reaches approximately 10mL, remove from the water bath, rinse the snyder column with approximately 1mL of methylene chloride, and allow the apparatus to drain and cool for at least 10 minutes. If the sample will not concentrate to a 10mL volume, consult your supervisor for instructions. Sign and date the KD portion of the extraction log.

8.2.18.4 Remove the synder column.

8.2.18.5 Transfer the contents from the KD assembly to a 20mL scintillation vial.

8.2.18.6 Rinse KD assembly three separate times, with 1-2mL of methylene chloride, and pour each rinse into the scintillation vial.

8.2.18.7 Place a preprinted sample ID label on scintillation vial.

8.2.19 Final Extract Concentration

8.2.19.1 Place the scintillation vial on the nitrogen blowdown apparatus. The flow of nitrogen should be such that it causes a slight ripple across the top of the sample without causing splashing of the sample. Concentrate the sample to approximately 1mL.

8.2.19.2 Transfer the sample to a 1.8mL Wheaton vial and put a preprinted sample ID label on the 1.8mL Wheaton vial. Rinse the scintillation vial with 0.5 mL of methylene chloride. Add the rinseate to the Wheaton vial.

8.2.19.3 Concentrate the sample to a final volume of 1mL. If the sample will not concentrate to a final volume of 1mL, inform supervisor. Record final volume on the extractions log and sign and date the blowdown portion of the extraction log.

8.2.20 Extract Submission

8.2.20.1 The Extraction log must be reviewed by the supervisor or designee before submitting sample extracts for analyses. After reviewing, sign and date the reviewed portion of the extraction log.

8.2.20.2 Submit extracts and extraction log to GC/MS Semivolatile (BNA) Dept. Sign and date the submitted portion of the extraction log.

8.2.20.3 Fill out the following fields on the GC/MS Extract Receipt Log, (Form-2): Date, Time, Sample ID, Client ID, and Extraction chemist Initials.

8.3 Method 3520C (Continuous Liquid - Liquid Extraction)

8.3.1 Pre-rinse all glassware and apparatus used with the extraction solvent.

8.3.2 Set up the Liquid-Liquid extractors by placing them in the (5 or 10)-port Liquid-Liquid extractor holder.

Note: Make sure that the stopcocks are in the closed position on the extractors.

8.3.3 Retrieve the appropriate number of 1000mL graduated cylinders, one for each sample + QC.

8.3.4 Retrieve the appropriate number of boiling flasks (500 mL) with 1 or 2 boiling chips in each, one for each sample + QC.

8.3.5 Install preprinted sample ID labels on Liquid-Liquid extractors an

8.3.6 d or boiling flask.

8.3.6 Extraction Solvent Addition

To each Boiling Flask add approximately 200 mL of methylene chloride; add approximately 250 mL of methylene chloride to each Liquid-Liquid extractor. Record the methylene chloride Lot # on the Extraction Log, in the comments section.

8.3.7 Shake sample bottle and pour sample into 1000 mL graduated cylinder. 1000mL of sample should be used. Record actual volume to nearest ½ graduated cylinder mark on the extraction log. Pour approximately 10-15ml of sample into a scintillation vial and take pH reading immediately and record in Extraction Log.

8.3.8 Pour sample from graduated cylinder into Liquid-Liquid extractor; care must be taken to ensure that the correct sample is poured into the correctly labeled extractor.

8.3.9 Continue steps 8.3.7 and 8.3.8 for each sample plus QC being extracted. For blanks and laboratory control spikes, Reagent water will be used as described in section 8.3.6.

Note: After all samples are poured out all remaining sample must be returned to the sample storage area and the internal chain of custody records must be filled to indicate that the remaining sample has been returned. If the entire sample was used for the extraction, then the Pitched Bottle Log must be filled out. This log is with the internal chain of custody records.

8.3.10 Surrogate Spike Addition

To each sample + QC add 500uL of surrogate spiking solution. Check to verify that you have the correct surrogate spiking solution, for the test code being performed, and that the surrogate spiking solution is not expired. Record surrogate spiking solution ID #, the spiking of each sample, and sample spike verification (second person) on Extraction Log. <u>The surrogate spiking solution is prepared and tested by the GC/MS Semivolatile analyses Dept. prior to use. When the solution is approximately 50 percent used up or expired, a new solution must be requested from the Semivolatile Dept.</u>

8.3.11 Laboratory Control Spike (LCS) Addition

To each Laboratory Control Sample (LCS) or duplicate (LCSD) add 500uL of LCS spiking solution. Check to verify that you have the correct LCS spiking solution, for the test code being performed, and that the LCS spiking solution is not expired. Record LCS spiking solution ID #, the spiking of LCS samples, and

sample spike verification (second person) on Extraction Log. This is normally the same solution as the Matrix Spike Solution and is recorded in the same location on the Extraction Log. When performing method 625, addition of all client specified compounds of interest is required. The laboratory control spiking solution is prepared and tested by the GC/MS Semivolatile analyses Dept. prior to use. When the solution is approximately 50 percent used up or expired, a new solution must be requested from the Semivolatile Dept.

8.3.12 Matrix Spike (MS) Addition

To each Matrix Spike Sample (MS) or duplicate (MSD) add 500uL of MS spiking solution. Check to verify that you have the correct MS spiking solution, for the test code being performed, and that the MS spiking solution is not expired. Record the MS spiking solution ID #, the spiking of MS samples, and sample spike verification (second person) on Extraction Log. This is normally the same solution as the Laboratory Control Spike Solution. When performing method 625, all client specified compounds of interest are required. The matrix spike spiking solution is prepared and tested by the GC/MS Semivolatile analyses Dept. prior to use. When the solution is approximately 50 percent used up or expired, a new solution must be requested from the Semivolatile Dept.

8.3.13 Initial pH Adjustment — If samples are to have both Base/neutral and Acid or Base/neutral only compounds tested continue with 8.3.9.1, and skip section 8.3.9.2. If samples are for Acid Extraction only (MS505 test code) then skip section 8.3.10.1 and start with section 8.3.10.2.

8.3.13.1 Base/Neutral pH adjustment — To each sample + QC slowly add (10N sodium hydroxide) until a pH value greater than 11 is constant by testing with narrow range pH paper (7 - 11). Sample should be stirred with a glass- stirring rod, then test the pH and again stir the sample and check the pH again, to verify proper pH. Record pH change on Extraction Log under Base pH confirmation and note pH change with >11.

Note: Extreme care must be taken when adding the 10N sodium sulfate, as to make certain that no solution and or sample gets into the evaporation arm of the extractor.

8.3.13.2 Acid pH adjustment — To each sample + QC slowly add (1:1) sulfuric acid until a pH value less than 2 is constant by testing with narrow range pH paper (1 - 6). Sample should be stirred with a glass - stirring rod, then test the pH and again stir the sample and check the pH again to verify proper pH. Record pH change on Extraction Log under Acid pH confirmation and note pH change with <2.

Note: Extreme care must be taken when adding the (1:1) sulfuric acid, as to make certain that no solution and or sample gets into the evaporation arm of the extractor.

8.3.14 Sample Extraction

8.3.14.1 Assemble the boiling flask, the liquid/liquid extractor and the condenser on the automated 18 hr Liquid-Liquid extraction apparatus.

8.3.14.2 Open stopcock on the Liquid-Liquid extractor so that the extractor will work properly. (Solvent will start to siphon as distillation starts.

8.3.14.3 Check the Cool-flow water recirculator, if need turn on and inspect to ensure re-circulator is working.

8.3.14.4 Turn on the 18 hr. automated heating mantles and let samples extract.

8.3.14.5 After samples have started extracting, check each Liquid-Liquid extractor to ensure that all joints are connected properly and extraction solvent is not leaking at any joint.

8.3.14.6 After the completion of the 18 Hr. extraction period the heating mantles will automatically shut off.

8.3.14.7 After the boiling flasks have cooled down, shut off the stopcock on the Liquid-Liquid extractors, and remove the water condensers.

8.3.14.8 If doing the Base/neutral extraction, Check the pH of extracted water to verify that pH is still <2, if not then readjust pH and repeat steps (8.3.14.1 - 8.3.14.7). If doing the Base extraction, Check the pH of extracted water to verify that pH is still >11, if not then readjust pH and repeat steps (8.3.14.1 - 8.3.14.7).

8.3.14.9 When the Acid extraction is completed and the Base Extraction is also needed on the samples, then proceed with section (8.3.14.10), other wise skip to section (8.3.15).

8.3.14.10 Second pH adjustment (Base) — To each sample + QC slowly add 10mL Sodium Hydroxide until a pH value greater 11 is constant by testing with narrow range pH paper (7-11). Sample should be stirred with a glass - stirring rod, then test the pH and again stir the sample and check the pH again to verify proper pH. Record pH change on Extraction Log under Base pH confirmation and note pH change with >11.

Note: Extreme care must be taken when adding the 10 N Sodium Hydroxide, as to make certain that no solution and or sample gets into the evaporation arm of the extractor.

8.3.14.11 After base pH is adjusted return to section (8.3.14) and continue the procedure through section (8.3.14.8).

8.3.14.12 Upon completion of the extraction, the extracted water and remaining methylene chloride in the liquid-liquid extractor is transferred to a separation vessel so that the solvent can be separated from the water.

8.3.14.13 The waste solvent is disposed as per Standard Operating Procedure "*Hazardous Waste Management Plan*", SWL-GA-114.

8.3.14.14 The extracted water is disposed in the normal manner.

8.3.14.15 The extract in the boiling flask is dried, by using sodium sulfate, prior to concentration, by either the KD or the Turbo-Vap method.

8.3.15 Initial Extract Concentration

8.3.15.1 KD method

8.3.15.2 Set up glass filter funnels, with S&S # 595 filter paper, and approximately 20 grams of Sodium Sulfate. Rinse the filer paper and sodium sulfate with methylene chloride. Record the Sodium Sulfate lot # on the Extraction Log, in the comment section.

8.3.15.3 Attach a concentrator tube to a KD flask and rinse both with methylene chloride.

8.3.15.4 Place a glass funnel containing sodium sulfate (previously prepared) above the KD assembly.

8.3.15.5 Slowly pour the solvent from the boiling flask into the funnel.

8.3.15.6 Rinse the boiling flask with approximately 10mL of the methylene chloride. Repeat this process two additional times. Each time pouring the rinses through the Na_2SO_4 and collecting in the KD.

8.3.15.7 After the solvent has been transferred to the KD flask, remove the funnel. Add two clean boiling chips to the KD

8.3.15.8 Attach a three-ball Snyder column to the KD. Prewet the column with approximately 1mL of methylene chloride.

8.3.15.9 Place the KD assembly in a hot water bath (not above 65°C) so that the entire lower rounded surface of the KD assembly is immersed in the water. The position of the KD assembly in the water bath and the water bath temperature should be such that the concentration is completed in 45 - 60 minutes. The balls of the Snyder column should actively chatter and the chambers should not flood with condensed solvent.

8.3.15.10 When the apparent volume of liquid reaches approximately 10mL, remove from the water bath, rinse the snyder column with

approximately 1mL of methylene chloride, and allow the apparatus to drain and cool for at least 10 minutes. If the sample will not concentrate to a 10mL volume, consult your supervisor for instructions. Sign and date the KD portion of the extraction log.

8.3.15.11 Remove the synder column.

8.3.15.12 Transfer the contents from the KD assembly to a 20mL scintillation vial.

8.3.15.13 Rinse KD assembly three separate times, with 1 - 2 mL of methylene chloride, and pour each rinse into the scintillation vial.

8.3.15.14 Place a preprinted sample ID label on scintillation vial.

8.3.16 Turbo-Vap method

8.3.16.1 Set up glass filter funnels, with S&S # 595 filter paper, and approximate 20 grams of Sodium Sulfate. Rinse the filer paper and sodium sulfate with methylene chloride. Record the Sodium Sulfate lot # on the Extraction Log, in the comment section.

8.3.16.2 Place a glass funnel (previously prepared) in the Zymark tube.

8.3.16.3 Slowly pour the solvent from the boiling flask into the funnel.

8.3.16.4 Rinse the boiling flask with three 10mL portions of the methylene chloride. Pour these rinses through the Na_2SO_4 and collect all in the Zymark tube.

8.3.16.5 After the solvent has been transferred to the Zymark tube, remove the funnel and place the zymark tube into the zymark concentrator.

8.3.16.6 Concentrate the sample to a 1mL volume, rinsing the walls of the Zymark tube with 1 to 2mL of methylene chloride several times during the concentration.

Note: When the volume of the extract reaches the 1 mL or 0.5mL mark according to the Turbovap used, a sensor will sound a long beep.

8.3.16.7 Transfer the sample to a 1.8 mL Wheaton vial and put a preprinted sample ID labels on the 1.8 mL Wheaton vial. Sign and date the KD portion of the extraction log noting from T/vap.

8.3.16.8 The extract is now ready for final concentration, proceed to section (8.3.17.3).

8.3.17 Final Extract Concentration

8.3.17.1 Place the scintillation vial on the nitrogen blowdown apparatus. The flow of nitrogen should be such that it causes a slight ripple across the top of the sample without causing splashing of the sample. Concentrate the sample to about 1 mL

8.3.17.2 Transfer the sample to a 1.8mL Wheaton vial and put a preprinted sample ID label on the 1.8mL Wheaton vial. Rinse the scintillation vial with 0.5 mL of methylene chloride. Add the rinsate to the Wheaton vial.

8.3.17.3 Concentrate the sample to a final volume of 1mL If the sample will not concentrate to a final volume of 1mL inform supervisor. Record the final volume on the extraction log, then sign and date the blowdown portion of the extraction log.

8.3.18 Extract Submission

8.3.18.1 The Extraction log must be reviewed by supervisor or a designee before submitting sample extracts for analyses. After review, sign and date the reviewed portion of the extraction log.

8.3.18.2 Submit extracts and extraction log to GC/MS Semivolatile (BNA) Dept. Sign and date the submitted portion of the extraction log.

8.3.18.3 Fill out the following fields on the GC/MS EXTRACT RECEIPT LOG, (FORM-2), Date, Time, Sample ID, Client ID, and Extraction Chemist Initials.

8.4 Method 3550B (Extraction of B/NA Fractions from Soil/Sediments) (Low Level)

8.4.1 Retrieve soil samples from sample storage, and fill out sample internal chain of custody records before removing samples from sample storage area.

8.4.2 Retrieve soil extraction log book and fill out pertinent information: Client, Date received, Extraction Date, SWLO Sample ID, client sample ID, Extraction type, matrix, etc.

8.4.3 Prepare preprinted extraction labels for all samples and QC, each sample should have 4 preprinted labels to be used during extraction and final blowdown.

8.4.4 Retrieve sufficient amount of 400 mL beakers, one for each sample + QC, and place a preprinted sample ID label on each beaker.

8.4.5 Homogenize sample before taking subsamples. This is done, by carefully removing any large stones, sticks, and then decanting any water at the top of the sample before mixing. Then mix the sample in the sample container by using a stainless steel spatula.

8.4.6 Make pH determination of each sample (see section 8.6.3 for procedure), if pH if < 5 or > 9 pH units, inform the supervisor of the pH on these samples. Record pH on the Extraction Log for each sample.

8.4.7 Determine the moisture content of each sample (see section 8.7 for procedure). Record % moisture on the Extraction Log for each sample.

8.4.8 Calibrate the balance before weighing samples if balance was not previously calibrated for the day. Check Zone Log Book on procedure for calibration balance.

8.4.9 Weigh 30 grams of the sample to the nearest 0.1gram into a 400 ml beaker. Record the weight on the Extraction Log for each sample + QC. For blanks and laboratory control spikes, pre-extracted white sand will be used for the 30gram sample weight.

8.4.10 Add approximately 60 grams of anhydrous sodium sulfate, to the 400mL beaker and mix well with a glass rod or stainless steel spatula. Record the Sodium Sulfate lot # on the Extraction Log, in the comment section.

8.4.11 Continue doing 8.4.9 and 8.4.10 for each sample plus QC being extracted.

8.4.12 Surrogate Spike Addition — To each sample + QC add 0.5mL of surrogate spiking solution. Check to verify that you have the correct surrogate spiking solution, for the test code being performed, and that the surrogate spiking solution is not expired. Record surrogate spiking solution ID #, the spiking of each sample, and sample spike verification (second person) on Extraction Log.

Note: If the samples are to go through the GPC cleanup step, add 500uL of surrogate spiking solution. The surrogate spiking solution is prepared and tested by the GC/MS Semivolatile analyses Dept. prior to use. When the solution is approximately 50 percent used up or expired, a new solution must be requested from the Semivolatile Dept. Final volume of extract is 0.5ml. If no GPC is performed, then final volume of extract is 1.0ml.

8.4.13 Laboratory Control Spike (LCS) Addition — To each Laboratory Control Sample (LCS) or duplicate (LCSD) add 500uL of LCS spiking solution. Check to verify that you have the correct LCS spiking solution, for the test code being performed, and that the LCS spiking solution is not expired. Record the LCS spiking solution ID #, the spiking of LCS samples, and sample spike verification (second person) on Extraction Log. This is normally the same solution as the Matrix Spike Solution and is recorded in the same location on the Extraction Log.

Note: If the samples are to go through the GPC cleanup step, add 500uL of LCS spiking solution. The laboratory control spiking solution is prepared and tested by the GC/MS Semivolatile analyses Dept. prior to use. When the solution is approximately 50 percent used up or expired, a new solution must be requested from the Semivolatile Dept. Final volume of extract is 0.5ml. If no GPC is performed, then final volume of extract is 1.0ml.

8.4.14 Matrix Spike (MS) Addition — To each Matrix Spike Sample (MS) or duplicate (MSD) add 500uL of MS spiking solution. Check to verify that you have the

correct MS spiking solution, for the test code being performed, and that the MS spiking solution is not expired. Record the MS spiking solution ID #, the spiking of MS samples, and sample spike verification (second person) on the Extraction Log. This is normally the same solution as the Laboratory Control Spike Solution.

Note: If the samples are to go through the GPC cleanup step, add 500uL of MS spiking solution. The matrix spike spiking solution is prepared and tested by the GC/MS Semivolatile analyses Dept. prior to use. When the solution is approximately 50 percent used up or expired, a new solution must be requested from the Semivolatile Dept. Final volume of extract is 0.5ml. If no GPC is performed, then final volume of extract is 1.0ml.

Note: After all samples are weighted out, all remaining sample must be returned to the sample storage area, and the internal chain of custody records must be filled to indicate that the remaining sample has been returned. If the entire sample was used for the extraction, then the Pitched Bottle Log must be filled out. This log is with the internal chain of custody records.

8.4.15 Extraction Solvent Addition

Soon after adding sodium sulfate, add to each beaker 100 ml of 1:1 methylene chloride/acetone Record the methylene chloride Lot # and the Acetone Lot # on the Extraction Log, in the comments section.

8.4.16 Sample Sonication

8.4.16.1 Set up glass funnels, with S&S # 595 filter paper, and approximately 10 to 20 grams of Sodium Sulfate, in 8-port soil extraction funnel holder (top level). Record the Sodium Sulfate lot # on the Extraction Log, in the comment section.

8.4.16.2 Set up Kuderna Danish concentrator assembly with boiling chips in the 8-port soil funnel holder, (lower level).

8.4.16.3 Install preprinted sample ID labels on kuderna Danish concentrator.

8.4.16.4 Sonicate the samples using the ¾" probe held about ½" below the liquid surface but ½" above the sediment level.

8.4.16.5 Use full power and a 50% pulse for 3 minutes.

8.4.16.6 Stir the extract with the stainless steel spatula, then decant and filter the extract through a filter paper in a glass funnel, collecting the extract in the KD.

Note: Extreme care must be taken to ensure that the correct sample is put into properly labeled KD, as not to switch samples.

8.4.16.7 Add another 100 ml of the 1:1 methylene chloride/acetone to the 400mL beakers containing the samples, stir the sample with the

stainless steel spatula and repeat sections (8.4.16.4 - 8.4.16.6). Repeat this section twice.

8.4.16.8 Rinse beaker and filtered funnel with approximately 50mL of methylene chloride.

8.4.16.9 Check Sodium Sulfate, if the sodium sulfate has hardened then the methylene chloride/acetone extract must be re-dried using new sodium sulfate. This must be done before the sample extract is concentrated. When the extract is deemed to be dry then proceed to section (8.4.16.10).

8.4.16.10 Initial Extract Concentration

- Attach a three-ball Snyder column to the KD. Prewet the column with approximately 1mL of methylene chloride.
- Place the KD assembly in a hot water bath (not to exceed 75°C) so that the entire lower rounded surface of the KD assembly is immersed in the water. The position of the KD assembly in the water bath and the water bath temperature should be such that the concentration is completed in 45 - 60 minutes. The balls of the Snyder column should actively chatter and the chambers should not flood with condensed solvent.
- When the apparent volume of liquid reaches approximately 10mL, remove from the water bath, rinse the snyder column with approximately 1mL of methylene chloride, and allow the apparatus to drain and cool for at least 10 minutes. If the sample will not concentrate to a 10mL volume, consult your supervisor for instructions. Sign & date the KD portion of the extraction log.
- Remove the synder column.
- Transfer the contents from the KD assembly to a 20mL scintillation vial.
- Rinse KD assembly three separate times, with 1-2mL of methylene chloride, and pour each rinse into the scintillation vial.
- Place a preprinted sample ID label on scintillation vial.
- Place the scintillation vial on the nitrogen blowdown apparatus. The flow of nitrogen should be such that it causes a slight ripple across the top of the sample without causing splashing of the sample. Concentrate the sample to 10mL.

- Filter each sample extract + QC through an Acrodics, 0.45 micron, (Gelman or equivalent), using 10mL PP+PE sterile disposable syringes, and place filtered extract back into a labeled 20mL scintillation vial.
- If no GPC is necessary then proceed to section 8.4.18. If sample extracts are to be GPC cleaned-up, then send extracts to GPC area and fill out the GPC portion of the extraction log.

8.4.17 Secondary Extract Concentration

8.4.17.1 After sample extracts have undergone GPC clean - up, the extracts need to be re-concentrated by use of a turbo-vap concentrator.

8.4.17.2 Transfer the extract from the GPC collection vial into a Zymark tube and put a preprinted sample ID label on the Zymark tube.

8.4.17.3 Rinse the GPC collection vial with approximately 10mL of the methylene chloride. Pour the rinse into the Zymark tube.

8.4.17.4 After the solvent has been transferred to the Zymark tube, place the zymark tube in the zymark concentrator (turbovap).

8.4.17.5 Concentrate the sample to a 1mL volume, rinsing the walls of the Zymark tube with 1 to 2mL of methylene chloride several times during the concentration process.

8.4.17.6 Transfer the sample to a 1.8mL Wheaton vial and place a preprinted sample ID label on the 1.8mL Wheaton vial. Sign and date the KD portion of the extraction log noting TV for turbovap.

8.4.17.7 Proceed to section 8.4.18.3 for final extract concentration.

8.4.18 Final Extract Concentration

8.4.18.1 Place the scintillation vial on the nitrogen blowdown apparatus. The flow of nitrogen should be such that it causes a slight ripple across the top of the sample without causing splashing of the sample. Concentrate the sample to about 1mL.

8.4.18.2 Transfer the sample to a 1.8mL Wheaton vial and put a preprinted sample ID labels on the 1.8mL Wheaton vial. Rinse the scintillation vial with 0.5mL of methylene chloride. Add the rinsate to the Wheaton vial.

8.4.18.3 Concentrate the sample to a final volume of 0.5mL. If the sample will not concentrate to a final volume of 0.5mL inform supervisor. Record final volume on the extractions log and sign and date the blowdown portion of the extraction log.

8.4.19 Extract Submission

8.4.19.1 The Extraction log must be reviewed by the supervisor or a designee before submitting the sample extracts for analyses. After the extraction log is reviewed, sign and date the review portion of the extraction log.

8.4.19.2 Submit extracts and extraction log to GC/MS Semivolatile (BNA) Dept. Sign and date the submitted portion of the extraction log.

8.4.19.3 Fill out the following fields on the GC/MS EXTRACT RECEIPT LOG, (FORM-2), Date, Time, Sample ID, Client ID, and Extraction Chemist Initials.

8.5 Method 3550B Extraction of BNA Fractions from Soil/Sediments (Medium/High Level)

8.5.1 Retrieve soil samples from sample storage, and fill out sample internal chain of custody records before removing samples from sample storage area.

8.5.2 Retrieve soil extraction log book and fill out pertinent information: Client, Date received, Extraction Date, SWLO Sample ID, client sample ID, Extraction type, matrix, etc.

8.5.3 Prepare preprinted extraction labels for all samples and QC, each sample should have 4 preprinted labels to be used during extraction and final blowdown.

8.5.4 Retrieve sufficient amount of 20mL scintillation vials, one for each sample + QC, and place a preprinted sample ID label on each vial.

8.5.5 Homogenize sample before taking subsamples. This is done, by carefully removing large stones, sticks, and then decanting any water at the top of the sample before mixing. Mix the sample in the sample container by using a stainless steel spatula.

8.5.6 Make pH determination of each sample (see section 8.6.3 for procedure), if pH if < 5 or > 9 pH units, inform the supervisor of the pH on these samples. Record the pH on the Extraction Log for each sample.

8.5.7 Determine the moisture content of each sample (see section 8.7 for procedure). Record % moisture on the Extraction Log for each sample.

8.5.8 Calibrate the balance before weighing samples if balance was not previously calibrated for the day. Check Zone Log Book on procedure for calibration balance.

8.5.9 Weigh 1 gram of the sample to the nearest 0.05 grams into a 20mL scintillation vial. Record the weight on the Extraction Log for each sample + QC. For blanks and laboratory control spikes, pre-extracted white sand will be used for the 1gram sample weight.

8.5.10 Add approximately 2 grams of anhydrous sodium sulfate, to the scintillation vial and mix well with a glass rod or stainless steel spatula. Record the Sodium Sulfate lot # on the Extraction Log, in the comment section.

8.5.11 Continue steps 8.5.9 and 8.5.10 for each sample plus QC being extracted.

Note: After all samples are weighted out, all remaining sample must be returned to the sample storage area, and the internal chain of custody records must be filled to indicate that the remaining sample has been returned. If the entire sample was used for the extraction, then the Pitched Bottle Log must be filled out. This log is with the internal chain of custody records.

8.5.12 Surrogate Spike Addition

To each sample + QC add 0.5 mL of surrogate spiking solution. Check to verify that you have the correct surrogate spiking solution, for the test code being performed, and that the surrogate spiking solution is not expired. Record surrogate spiking solution ID #, the spiking of each sample, and the sample spike verification (second person) on Extraction Log.

Note: If the samples are to go through the GPC cleanup step, add 500uL of surrogate spiking solution. The surrogate spiking solution is prepared and tested by the GC/MS Semivolatile analyses Dept. prior to use. When the solution is approximately 50 percent used up or expired, a new solution must be requested from the Semivolatile Dept. Final volume of extract is 0.5ml. If no GPC is performed, then final volume of extract is 1.0ml.

8.5.13 Laboratory Control Spike (LCS) Addition

To each Laboratory Control Sample (LCS) or duplicate (LCSD) add 500uL of LCS spiking solution. Check to verify that you have the correct LCS spiking solution, for the test code being performed, and that the LCS spiking solution is not expired. Record the LCS spiking solution ID #, the spiking of LCS samples, and sample spike verification (second person) on Extraction Log. This is normally the same solution as the Matrix Spike Solution and is recorded in the same location on the Extraction Log.

Note: If the samples are to go through the GPC cleanup step, then add 500uL of LCS spiking solution. The laboratory control spiking solution is prepared and tested by the GC/MS Semivolatile analyses Dept. prior to use. When the solution is approximately 50 percent used up or expired, a new solution must be requested from the Semivolatile Dept. Final volume of extract is 0.5ml. If no GPC is performed, then final volume of extract is 1.0ml.

8.5.14 Matrix Spike (MS) Addition

To each Matrix Spike Sample (MS) or duplicate (MSD) add 500uL of the MS spiking solution. Check to verify that you have the correct MS spiking solution, for the test code being performed, and that the MS spiking solution is not expired. Record the MS spiking solution ID #, the spiking of MS samples, and sample spike verification (second person) on Extraction Log. This is normally the same solution as the Laboratory Control Spike Solution.

Note: If the samples are to go through the GPC cleanup step, add 500uL of MS spiking solution. The matrix spike spiking solution is prepared and tested by the GC/MS Semivolatile analyses Dept. prior to use. When the solution is approximately 50 percent used up or expired, a new solution must be requested from the Semivolatile Dept. Final volume of extract is 0.5ml. If no GPC is performed, then final volume of extract is 1.0ml.

8.5.15 Extraction Solvent Addition

To each scintillation vial add the appropriate amount of methylene chloride solution. The appropriate amount will be such that with the addition of the spiking volumes added to each scintillation vial, for surrogate, laboratory control spike, and matrix spike, the total volume added to each scintillation vial is 10mL. Record the methylene chloride Lot # on the Extraction Log, in the comments section.

8.5.16 Sample Sonication

8.5.16.1 Sonicate the samples using the 4-prong microprobe held about ½" below the liquid surface but ½" above the sediment level.

8.5.16.2 Use full power and a 50% pulse for 3 minutes.

8.5.16.3 Retrieve a sufficient amount of 10 mL PP+PE sterile disposable syringes with Acrodics, 0.45 micron, (Gelman or equivalent.), to filter each sample extract + QC.

8.5.16.4 Add approximately 2 millimeters of glass wool to the inside of each disposable syringe.

8.5.16.5 Add approximately 2 millimeters of anhydrous sodium sulfate to the inside of each disposable syringe.

8.5.16.6 Stir the extract with the stainless steel spatula, and then decant the sample extract into the previously prepared disposable syringes (section 8.5.16.3 through 8.5.16.5).

8.5.16.7 Filter the extract through the Acrodics, 0.45 micron, (Gelman or equivalent.), and place filtered extract back into a labeled 20mL scintillation vial.

8.5.16.8 If no GPC is necessary then proceed to section 8.4.18. If sample extracts are to be GPC cleaned-up send extracts to GPC area and fill out the GPC portion of the extraction log.

8.5.16.9 After GPC is complete, proceed to section 8.4.17.

8.6 pH Determination

8.6.1 The pH meter is to be calibrated before use and calibration data recorded in the pH meter logbook.

8.6.2 Water samples

8.6.2.1 Shake the sample and transfer approximately 20ml to a clean scintillation vial.

8.6.2.2 Place the pH probe into the liquid and allow the meter to obtain a stable reading.

8.6.2.3 Record the pH reading on the extraction log, as soon as the sample is measured for extraction.

8.6.3 Soil samples

8.6.3.1 Homogenize the sample.

8.6.3.2 Weigh approximately 30 grams of sample into a clean polypropylene container.

8.6.3.3 Add 30ml of deionized water to the sample.

8.6.3.4 Mix sample and water with a stainless steel spatula.

8.6.3.5 Cover the polypropylene container with a polyethylene cap.

8.6.3.6 Allow the sample to shake for one hour on an automatic shaker.

8.6.3.7 After 1 hour, place the pH probe into the liquid and allow the meter to obtain a stable reading.

8.6.3.8 Record the pH reading on the sample data sheet and on the extraction log.

8.7 Moisture Determination

8.7.1 Use Southwest Laboratories Automated Percent Moisture Program.

8.7.2 Enter in Southwest Lab ID (episode #) for sample

8.7.3 Label (using a marker) the aluminum weighting pan (70 X 16 mm), with same ID as in 8.7.2.

8.7.4 Tare the aluminum weighting pan (70 X 16 mm) on the balance.

8.7.5 Weigh 5-10 grams of the sediment into the aluminum weighting pan (70 X 16mm).

8.7.6 Place samples into a 103 $\pm$ 2 degrees Celsius drying oven.

8.7.7 Dry samples over night or for a predetermined amount of time.

8.7.8 Remove samples from oven and allow the samples to cool at room temperature before weighing.

8.7.9 Reweigh the aluminum pans (containing the dried sample) on the balance using the automated percent moisture program.

8.7.10 Print out the percent moisture reports from the automated program and store in a 3 ring binder.

8.7.11 Record the percent moisture on the extraction log.

9.0 CALCULATIONS

9.1 Percent Moisture Calculation

$$\frac{(\text{wet weight of sample} - \text{dry weight of sample}) \times 100}{(\text{wet weight of sample})} = \text{Percent Moisture}$$

9.2 10 Normal Sodium Hydroxide Solution (10N)

9.2.1 Add 3 liter of reagent grade water to a 3 liter Erlenmeyer flask

9.2.2 Add stirring bar to 3 liter Erlenmeyer flask.

9.2.3 Add flask to stirring plate and start stirring.

9.2.4 Slowly add 1200 grams (400 grams per liter of water) of Sodium Hydroxide pellets (Fisher certified A.C.S.) to the Reagent grade water.

Note: Solution will start to heat up.

9.2.5 Allow solution to stir until all pellets have dissolved.

9.2.6 After solution has cooled to room temperature, the 10N Sodium Hydroxide solution is ready for use.

9.3 1:1 Sulfuric Acid Solution

9.3.1 Add 1 liter of reagent grade water to a 3 liter Erlenmeyer flask

9.3.2 Add stirring bar to 3 liter Erlenmeyer flask.

9.3.3 Add flask to stirring plate and start stirring.

9.3.4 Slowly add 1 liter of concentrated Sulfuric Acid (Fisher certified A.C.S. PLUS) or equivalent to the Erlenmeyer flask.

Note: Solution will start to heat up.

9.2.5 Allow solution to stir until all Sulfuric Acid has been added to the water.

9.2.6 After solution has cooled to room temperature, the 1:1 sulfuric acid solution is ready for use.

10.0 QUALITY ASSURANCE/QUALITY CONTROL

The GC/MS Semivolatile Dept monitors quality Assurance/Quality Control. This is accomplished by reviewing the extraction recoveries on the laboratory control spikes and surrogates.

11.0 WASTE DISPOSAL/POLLUTION PREVENTION

All organic extraction waste is to be disposed in accordance with the latest revision of "*Hazardous Waste Management Plan*", SOP SWL-GA-114.

12.0 METHOD PERFORMANCE

The GC/MS Semivolatile Dept monitors Method performance. This is accomplished by reviewing the extraction recoveries on the laboratory control spikes and surrogates.

13.0 REFERENCES

13.1 SW846 Revision 3 Dec. 1997 Methods: 3510C, 3520C, and Revision 2 Dec. 1997 Method 3550B.

13.2 USEPA Contract Laboratory Program, Statement of Work for Organic analysis, Multi-Media, Multi-concentration, OLMO3.2

13.3 Standard Operating Procedure "*Hazardous Waste Management Plan*", SWL-GA-114.

13.4 Standard Operating Procedure "*Laboratory Safety Plan*", SWL-GA-111.

14.0 DEFINITIONS

14.1 Sep. Funnel ..... Short for Separatory funnel used in the extraction of aqueous water samples.

14.2 SEP-F ..... Short for Separatory funnel used in the extraction of aqueous water samples.

14.3 cont. Ex ..... Short for continuous liquid-liquid extractor used in the extraction of aqueous water samples.

14.4 Liquid-Liquid..... Short for continuous liquid-liquid extractor used in the extraction of aqueous water samples.

14.5 Aqueous liquid..... A liquid which is soluble in water.

14.6 PPE..... Personnel Protective Equipment

14.7 Degradation..... The breaking down of a compound into different compounds.

14.8 QC ..... Quality Control, used in this SOP to indicate types of samples such as extraction blanks, laboratory control spikes, and matrix spikes.

14.9 LCS ..... Laboratory Control Spike

14.10 LCSD ..... Laboratory Control Spike Duplicate

14.11 MS ..... Matrix Spike

14.12 MSD ..... Matrix Spike Duplicate

14.13 (second person) ..... The person doing the spike verification for sample spiking.

14.14 KD ..... Kuderna Danish solvent concentrator apparatus

14.15 Ml ..... Milliliters

14.16 gm ..... Grams

14.17 ID ..... Identification

14.18 # ..... Number

14.19 1:1 ..... Represents a 50 percent volume to 50 percent volume ratio

14.20 S&S # 595 ..... Type of filter paper for Schleicher and Schuell

14.21 GPC ..... Gel Permeation Chromatography

15.0 ATTACHMENTS

Not applicable.

SOUTHWEST LABORATORY OF OKLAHOMA, INC. *and*
AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.

TM

Standard Operating Procedure

Purge & Trap Maintenance

Document No.: SWL-OG-118

Rev No. / Date: Rev. 1.1 — 08/24/99

APPROVALS	DATES
Procedure Prepared By	Date
Program Manager or Section Supervisor	Date
Laboratory Safety Officer	Date
Laboratory QA/QC Officer	Date

(Effective Date is 5 calendar days after the last signature above - QA/QC Officer)

Document Status

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PURGE & TRAP MAINTENANCE

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1.0 SCOPE AND APPLICATION

1.1 The purpose of this standard operating procedure is to provide guidelines for purge and trap instrument maintenance.

1.2 The purge & trap instruments require daily clean-up to ensure the sample analysis is accurate and precise.

2.0 METHOD SUMMARY

2.1 None.

3.0 HEALTH AND SAFETY

3.1 Care should be exercised as test tubes and vials are removed from the purge & trap instrument since they are glass and could be harmful if broken.

3.2 All test tubes and vials removed from the purge & trap instruments contain volatile standards or samples and are treated as potentially hazardous. Exposure to their contents is minimized.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING & STORAGE

4.1 None.

5.0 INTERFERENCE AND POTENTIAL PROBLEMS

5.1 Care should be taken to coordinate comments about sample analysis in the run logbook and clean-up activities asssociated with spcific purge locations..

6.0 EQUIPMENT/APPARATUS

6.1 Concentrators:Tekmar LSC2000, 3000, or equivalent

6.2 Autosamplers: Tekmar ALS2032, ALS2016, Precept; Varian Archon or equivalent

7.0 REAGENTS

7.1 Methanol

7.2 Reagent Water

8.0 PROCEDURE

8.1 Daily Maintenance (Tekmar ALS2032/2016)

8.1.1 Remove test tubes, and using a syringe, flush clean water through purge needles. Note any discrepancies in the previous night's run sequence.

8.1.2 Wipe off needles if soils were analyzed

8.1.3 Purge each chamber to clear residual water and/or soil.

8.1.4 If a sample contained target compounds or tentatively identified compounds at high levels, it may have contaminated the chamber and/or lines. Place a test tube in that position and "Step to Bake" with the Bake Gas Bypass "On" which means no gas flows through the chamber. This procedure will decrease the chances of soil getting into the sample return lines.

8.1.5 Verify non-soil needles keep a slight bend in them during the cleaning procedure. This prevents possible blockage that may occur during routine soil analysis.

8.2 Daily Maintenance (Tekmar Precept or Varian Archon)

8.2.1 Remove VOA vials from tray, noting any discrepancies in the previous night's run sequence.

8.2.2 Check the level of standard(s) in their vials and make new standards if necessary. Prime any new standards added to the Precept or Archon before analysis begins.

8.2.3 Check water level in independent water storage tanks and refill if necessary. Prime water line after refill.

8.2.4 Check nitrogen level in tank (used to pressurize Precept water tank) and replace if necessary.

8.2.5 Check water drain storage container and empty if necessary.

8.3 Preventive Maintenance

8.3.1 Little operator maintenance is required beyond daily clean-up.

8.3.2 The trap is changed as required by protocol or as it degrades and compounds are no longer trapped efficiently.

9.0 CALCULATIONS

9.1 None

10.0 QUALITY ASSURANCE/QUALITY CONTROL

10.1 None

11.0 WASTE DISPOSAL

11.1 Residual sample material and standards are discarded as directed by the Hazardous Waste Management Plan (SWL-GA-114, Rev. 3.0)

12.0 METHOD PERFORMANCE

12.1 None

13.0 REFERENCES

13.1 Hazardous Waste Management Plan (SWL-GA-114, Rev. 3.0)

14.0 DEFINITIONS

14.1 Terminology

Standard Operating Procedure (SOP): ..... Written narratives that describe step-by-step instructions and procedures specific to the laboratory.

GC/MS Run Log: ..... Log containing daily volatile analytical analyses. Maintained for each instrument.

Heating Pad Monitoring Log..... Log recording temperature verification of heating pads used in volatile soil analysis.

15.0 ATTACHMENTS

15.1 None

SOUTHWEST LABORATORY OF OKLAHOMA, INC.

Standard Operating Procedure

Liquid Chromatography Instrument Maintenance Log

Document No.: SWL-OL-103

Rev No. / Date: Rev. 1.1 — 04/23/98

APPROVALS	DATES
Procedure Prepared By	Date
Organic Program Manager	Date
Laboratory Safety Officer	Date
Laboratory QA/QC Officer	Date

(Effective Date is 5 calendar days after the last signature above - QA/QC Officer)

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LIQUID CHROMATOGRAPHY INSTRUMENT MAINTENANCE LOG

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1.0 SCOPE AND APPLICATION. This SOP defines the procedure for routine maintenance, scheduling and documentation for Liquid Chromatographs.

2.0 METHOD SUMMARY

2.1 Preventive maintenance is performed as scheduled or on a needed basis.

2.2 All of the performed maintenance is recorded in the maintenance log book. Each instrument has a specified maintenance log book.

3.0 HEALTH AND SAFETY

3.1 Manufacturer's specification should be followed when replacing any parts.

3.2 Always turn the power off to the instrument during any maintenance.

3.3 Some parts contain chemicals, and/or reagents which the toxicity or carcinogenicity of these reagents are not precisely defined. Therefore, these chemicals must be treated as a potential health hazard and exposure to them should be reduced to the lowest possible level by whatever means available. Refer to SWLO's Laboratory Safety Plan, SWL-GA-111.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE. N/A.

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

5.1 The parts that are exposed to the reagents should always be cleaned and be residue free during replacement.

5.2 When it is recommended, gloves should be worn to eliminate any possible contamination.

5.3 Follow the manufacturer's recommendation to eliminate any introduction of interference.

5.4 Helium should always be used as a degassing agent since other gases can not be used as a degassing agent.

6.0 EQUIPMENT/ APPARATUS

6.1 Recommended tools by the manufacturer.

6.2 Recommended solvents to clean the parts by the manufacturer.

7.0 REAGENTS

7.1 HPLC Grade Methanol (or equivalent).

7.2 (THF) Tetrahydrofuran-HPLC grade (or equivalent).

7.3 HPLC water (or equivalent).

8.0 PROCEDURE

8.1 Follow manufacturer's recommendation for each instrument regarding preventive maintenance.

8.2 The following preventive measurements should be followed to maximize instrument utilization.

8.2.1 Check the pump seals every three months or sooner according to utilization.

8.2.2 Replace the piston seals if any signs of shedding appears.

8.2.3 Check the piston sapphire rod for scratches. This can be performed most effectively by using a magnifying glass. If there are scratches on the piston sapphire rod, the rod should be replaced before any piston seal installation.

8.2.4 Check the columns for resolution and separation during analysis.

8.2.5 Check the instruments for contamination by analyzing instrument blanks periodically at the start of the analysis.

8.2.6 Observe any retention time shift that may occur during the analysis due to the instrument and/or column.

8.2.7 If the retention time shift persists, the following measurements should be taken.

- Check for visible leaks in the entire system while the pumps are operating.
- If the old column is being utilized, exchange the column with a newer column.
- Change the piston seals and rod if required.
- If the problem still persists, consult the supervisor for further investigation.

8.2.8 The UV lamp should be replaced every 1000 hours if it is required. However, the lamp can be utilized until the occurrence of the following problems:

- Baseline noise increases by 50%.
- Sign of spiking appears during analysis.
- Low illumination of the lamp can be checked through the reference and the sample

8.2.9 After installation of a new deuterium lamp, make a note of reference and a sample. This will be helpful in determining the lamp condition.

8.2.10 Some of the deuterium lamps are equipped with a timer which specifies the number of hours the lamps have been utilized.

8.2.11 When a new deuterium lamp is installed, allow the lamp to warm up for 2-3 hours before utilization of the detector.

8.2.12 Consult the manual on each instrument for the lamp replacement. If problems occur, consult the supervisor.

8.2.13 The instruments should be checked monthly for preventive maintenance measurements, and the preventive maintenance log sheets should be initialed for each instrument. Performed maintenance should be recorded on the HPLC maintenance log sheet.

8.2.14 When the instrument has been returned to the working status, follow up should be recorded on the HPLC maintenance log sheet.

8.2.15 All the solvents prior to use should be filtered in order to remove the particulate.

8.2.16 Degas the solvents for 5-10 minutes, and prime the pump prior to utilization.

9.0 CALCULATION. N/A.

10.0 QUALITY ASSURANCE/ QUALITY CONTROL. To eliminate contamination, manufacturer's specification should be followed for replacement of parts.

11.0 WASTE DISPOSAL. Refer to SWL-GA-114 SOP for waste disposal.

12.0 METHOD PERFORMANCE. N/A.

13.0 REFERENCES

13.1 Laboratory Quality Assurance Plan; SWL-GA-100.

13.2 Standard Operating Procedure for Preventative Maintenance, SWL-GA-127.

14.0 DEFINITIONS

14.1 HPLC: High Pressure Liquid Chromatography.

14.2 N/A: Not Applicable.

15.0 ATTACHMENTS

15.1 Attachment 1: An example of a Preventive Maintenance Log Sheet.

15.2 Attachment 2: An example of an HPLC Instrument Maintenance Log.

ATTACHMENT 1
PREVENTIVE MAINTENANCE LOG SHEET

Preventive Maintenance Log Sheet

SWL SOP Rev. # 1.0 - 08/ 13/97; Form ID: OL-103-PREV-F: Location/File ID: F:\SOP\OL\103-ATT1

PREVENTIVE MAINTENANCE SCHEDULE IN 12 WATERS									
MONTH	YEAR								
	1997	1998	1999	2000	2001	2002	2003	2004	2005
JAN.									
FEB.									
MARCH									
APRIL									
MAY									
JUNE									
JULY									
AUG.									
SEPT.									
OCT.									
NOV.									
DEC.									

1. Check the seals in the pumps every three months.
2. Check the columns for resolution and separation during analysis.
3. Check the instrument for contamination by analyzing the solvent blank periodically.
4. Observe any retention time shift that may occur due to instrument and/or column. Correct the problems accordingly.
5. Replace the UV lamp every 1000 hours if it is required or if there is a 50% increase in baseline noise.

ATTACHMENT 2
HPLC INSTRUMENT MAINTENANCE LOG

HPLC Instrument Maintenance Log

SWL SOP Rev. # 1.0 - 08/ 14/97; Form ID: OL-103-INSLOG-F: Location/File ID: F:\SOP\OL\103-ATT2

Description Of Maintenance	Date Performed	Initial	Follow up Date

SOUTHWEST LABORATORY OF OKLAHOMA, INC.

HPLC Instrument Maintenance Log

LC5

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Preventive Maintenance Log Sheet

Prev. Maint. Log Sheet (OL103Att.wb1) SWL-OL-103 Rev. 1.1

PREVENTIVE MAINTENANCE SCHEDULE LC5							
MONTH	YEAR						
	1999	2000	2001	2002	2003	2004	2005
JAN.							
FEB.							
MARCH							
APRIL							
MAY							
JUNE							
JULY							
AUG.							
SEPT.							
OCT.							
NOV.							
DEC.							

Check the seals in the pumps every three months.
Check the columns for resolution and separation during analysis.
Check the instrument for contamination by analyzing the solvent blank periodically.
Observe any retention time shift that may occur due to instrument and/or column. Correct the problems accordingly.
Replace the UV lamp every 1000 hours if it is required or if there is a 50% increase in baseline noise.

HPLC Instrument Maintenance Log

HPLC Instr. Maint. Log (OL103Att.wb1) SWL-OL-103 Rev. 1.1

Description of Maintenance	Date Performed	Initial	Follow-up Date

SOUTHWEST LABORATORY OF OKLAHOMA, INC. and
AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.

TM

Standard Operating Procedure

Nitroaromatics and Nitramines by HPLC, SW846 Method 8330

Document No.: SWL-OL-200

Rev No. / Date: Rev. 6.0 — 09/27/00

OFFICIAL COPY RED STAMP INDICATES ORIGINAL

APPROVALS	DATES
Procedure Prepared By	9-28-00 Date
Program Manager or Section Supervisor	9-10-3-00 Date
Laboratory Safety Officer	10/6/2000 Date
Laboratory QA/QC Officer	10-5-00 Date

(Effective Date is 5 calendar days after the last signature above - QA/QC Officer)

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NITROAROMATICS AND NITRAMINES BY HPLC, SW846 METHOD 8330

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1.0 SCOPE AND APPLICATION

1.1 This method (based on SW-846 Method 8330) is used to determine the concentration of the explosive residues in water, soil or sediment/s listed in Table 1.

1.2 This SOP has 1 deviation from Method 8330. Method 8330 calls for calibration standards to be made daily. SWLO uses calibration standards for 6 months (see Section 7.3.)

Table 1

COMPOUND	ABBR.	CAS Number
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine	HMX	2691-41-0
Hexahydro-1,3,5-trinitro-1,3,5-triazine	RDX	121-82-4
1,3,5-Trinitrobenzene	TNB	99-35-4
1,3-Dinitrobenzene	DNB	99-65-4-0
Methyl-2,4,6-trinitrophenylnitramine	Tetryl	479-45-8
Nitrobenzene	NB	98-95-3
2,4,6-Trinitrotoluene	TNT	18-96-7
4-amino-2,6-dinitrotoluene	4ADNT	1946-51-0
2-amino-4,6-dinitrotoluene	2ADNT	35572-78-2
2,6-Dinitrotoluene	26DNT	606-20-2
2,4-Dinitrotoluene	24DNT	121-14-2
o-Nitrotoluene	2NT	88-72-2
p-Nitrotoluene	4NT	99-99-0
m-Nitrotoluene	3NT	99-08-1
SURROGATE		
3,4-Dinitrotoluene	34DNT	610-39-9
* Chemical Abstracts Service Registry number		

Table 2
Extended Target List
(see section 8.1 for required modifications to chromatographic conditions)

COMPOUND	ABBR.	CAS Number
Pentaerythritol tetranitrate	PETN	75-11-5

1.3 All of these compounds are either used in the manufacturing of explosives or are the degradation products of compounds used for that purpose. When making stock solutions for calibration, treat each compound as if it were extremely explosive.

1.4 The practical quantitation limits (PQLs) of target analytes are presented in Table 3. The PQLs are based on the low point (L1) in the initial calibration represented in Table 4, the amount of sample extracted, and the volume of the extract.

1.5 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

explosive materials. Each analyst must demonstrate the ability to generate acceptable results with this method.

1.6 Method Detection Limits (MDLs) are generated according to SWL-GA-113, see "red book" for current MDLs.

Table 3
Practical Quantitation Limits

Compound	Low Level Water (ug/L)	High Level Water (ug/L)	Soil (ug/Kg)
HMX	0.65	100	500
RDX	0.65	100	500
TNB	0.65	100	500
DNB	0.65	100	500
TETRYL	0.65	100	500
NB	0.65	100	500
TNT	0.65	100	500
24DNT	0.65	100	500
26DNT	0.65	100	500
2NT	0.65	100	500
3NT	0.65	100	500
4NT	0.65	100	500
2ADNT	0.65	100	500
4ADNT	0.65	100	500

Table 4
Calibration Standards (µg/L)

Compound	L1	L2	L3	L4	L5
HMX	0.05	0.2	0.8	3.2	12.8
RDX	0.05	0.2	0.8	3.2	12.8
TNB	0.05	0.2	0.8	3.2	12.8
DNB	0.05	0.2	0.8	3.2	12.8
TETRYL	0.05	0.2	0.8	3.2	12.8
NB	0.05	0.2	0.8	3.2	12.8
TNT	0.05	0.2	0.8	3.2	12.8
4ADNT	0.05	0.2	0.8	3.2	12.8
2ADNT	0.05	0.2	0.8	3.2	12.8
26DNT	0.05	0.2	0.8	3.2	12.8
24DNT	0.05	0.2	0.8	3.2	12.8
2NT	0.05	0.2	0.8	3.2	12.8
4NT	0.05	0.2	0.8	3.2	12.8
3NT	0.05	0.2	0.8	3.2	12.8
34DNT (S)	0.05	0.2	0.8	3.2	12.8

2.0 METHOD SUMMARY

2.1 Aqueous Samples:

2.1.1 High level water (Direct Injection Method)

Samples are diluted 2X (v/v) with acetonitrile, separated on a Carbosorb reverse phase column (or equivalent), determined at 254nm, and confirmed on a Zorbax SB-CN column (or equivalent).

2.1.2 Low level water (Salting out):

The samples are extracted using NaCl and acetonitrile. The extracts are diluted 2X (v/v) with 0.5 % calcium chloride/water, separated on a Carbosorb reverse phase column (or equivalent), determined at 254 nm, and confirmed on a Zorbax SB-CN column (or equivalent).

2.2 Soil and sediment samples are extracted using acetonitrile in an ultrasonic bath. The extracts are filtered and diluted 2X (v/v) with 0.5 % calcium chloride/water.

3.0 HEALTH AND SAFETY

3.1 Standard precautionary measures used for handling other toxic organic compounds should be employed for safe handling of the analytes targeted by Method 8330.

3.2 Refer to SWL-GA-111, "*Laboratory Safety Plan.*"

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

4.1 Grab samples must be collected and stored in glass containers. Follow conventional sampling procedures.

4.2 Samples must be stored in the dark at 4 ± 2°C from the time of collection prior to analysis. Soil or sediment samples must be extracted within 14 days of the collection, and analyzed within 40 days of extraction. Water samples must be extracted within 7 days of collection, and analyzed within 40 days of extraction. High concentration water samples must be analyzed within 14 days of extraction

4.3 Soil and sediment samples after collection should be air dried at room temperature or colder to constant weight. While it is possible to analyze wet soil samples, it is much more difficult to obtain a homogeneous subsample on a wet sample. If wet soil samples are to be analyzed, a moisture determination must be made on a separate subsample.

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

5.1 Tetryl decomposes rapidly in methanol/water solutions, as well as with heat and light. All aqueous samples expected to contain Tetryl should be diluted with acetonitrile prior to filtration. All samples expected to contain Tetryl should not be exposed to temperatures above room temperature.

5.2 Salt present in acetonitrile extracted in the last step of the low concentration water (salting out) procedure produces interference peaks at the beginning of the chromatogram.

6.0 EQUIPMENT / APPARATUS

6.1 HPLC System

6.1.1 Autosampler

6.1.2 Pump - high-pressure pump capable of 6000 psi at 10 ml/min.

6.1.3 Column - Carbosorb, 5um, 250mm x 4.6mm ID (or equivalent) for quantitation
Zorbax SB-CN, 5um, 250mm x 4.6mm ID (or equivalent) for confirmation

6.1.4 Column Heater

6.1.5 Detector - Variable UV. Programmable UV is required when PETN is added to the target list.

6.1.6 Data System - Hewlett Packard Enviroquant.

6.2 Materials

6.2.1 Filters – Gelman Accuspec CR 0.45 µm filters for filtering extracts. or equivalent

6.2.2 Glass Pipettes – Class A, 10mL, 5mL, 4mL, 2mL, and 1mL.

6.2.3 Glass Vials – 1.5 mL, 12mL, 40ml, Teflon lined screw cap or equivalent.

6.2.4 Volumetric flasks – Class A - 10mL, 20mL, 50mL, 100mL, 200mL, 250mL

6.2.5 Rainin Pipettor – 10-100 µL, 100–1000 µL, and 1-10mL.

7.0 REAGENTS

7.1 HPLC grade chemicals shall be used in all tests. 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. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lowering the accuracy of the determination.

7.1.1 Acetonitrile, CH_3CN – HPLC grade.

7.1.2 Methanol, CH_3OH – HPLC grade.

7.1.3 Water, H_2O – Millipore (or equivalent).

7.2 Stock Standard Solutions. Certified standard mixes obtained from a vendor that contain the target analytes.

7.3 Working Calibration Standards.

Prepare 5 working calibration standards by diluting the stock standards with 50:50 (v/v) with a 5 g/L (0.5%) Calcium Chloride/water and acetonitrile. The low standard is at the PQL. Calibration verifications will be tested daily against the 5 point calibration. As

long as the response has not deteriorated. the working calibration standards may be used up to 6 months.

7.4 LCS (Laboratory Control Spike) and LCSD (Laboratory Control Spike Duplicate)

LCS are spiked with the analytes of interest at the midpoint level of the calibration curve.

7.5 MS (Matrix Spike) and MSD (Matrix Spike Duplicate)

MS and MSD are spiked with the analytes of interest at the midpoint level of the calibration curve.

7.6 Surrogate

34DNT, the analyte of non-interest used for the surrogate. is spiked at the midpoint level of the calibration curve.

7.7 Eluent

7.7.1 To prepare 1 liter 50% Methanol/H2O for the mobile phase. add 500ml of methanol to 500mL of HPLC water.

7.7.2 To prepare 1 liter of 50% Acetonitrile/H2O, add 500ml of Acetonitrile to 500ml of HPLC water.

7.7.3 The eluent should be filtered through a 0.45 µm filter subsequent to use.

7.7.4 The eluent should have helium continuously bubbled through the solution in order to remove air.

7.7.5 Prepare 5% calcium chloride/water by adding 5g of the solute to 1000 mL of HPLC water. Stir the solution on the stirring plate until all of the Calcium chloride has been dissolved. Place the solution in the refrigerator at 4°C if the solution is not used.

8.0 PROCEDURES

8.1 Chromatographic Conditions

8.1.1 Carbosorb quantitation column

Mobile Phase:	50/50 (v/v) methanol/HPLC water
Flow rate:	0.8 mL/min
Injection volume:	200 µL
Temperature:	28.0° C
UV Detector:	254 nm. If PETN is added to the target list, it is necessary to change the wavelength to 210 nm at approximately 19 minutes (before the 2NT peak) and to increase the run time by approximately 2 minutes.

8.1.2 Zorbax SB-CN confirmation column

Mobile Phase:	35/65 (v/v) methanol/HPLC water
Flow rate:	1.5 mL/min
Injection volume:	200 µL
Temperature:	30.0° C
UV Detector:	254 nm. If PETN is added to the target list, it is necessary to change the wavelength to 210 nm at approximately 17 minutes (after the tetryl peak) and to increase the run time by approximately 10 minutes.

8.2 Calibration of HPLC

8.2.1 Following SW-846, method 8330, the initial calibration standard at the minimal five- concentration is injected only one time. The average %RSD of the calibration factors should be ≤20%. Use the mean calibration factors for all calculations as long as the calibration verification described below meet specifications. Immediately following the injection of the initial calibration, an initial calibration verification (ICV), obtained from a different vendor than the initial calibration standards, is injected. The concentration of the ICV is equal to the mid level standard.

8.2.2 Calibration Factor (CF): 5-Level

$$CF = \frac{\text{Peak Area of Standard}}{\text{Concentration (ug/L)}}$$

$$\overline{CF} = \Sigma \frac{CF}{n}$$

$$\%RSD = \frac{STD \times 100}{\overline{CF}}$$

Where:

$\overline{CF}$ = Mean of the calibration factor
n = (5) Total number of calibration levels
STD = Standard deviation of the calibration factors
CF = Calibration factor

8.2.3 A calibration verification using the mid-level standard should be injected at the beginning of an analysis day and the retention times should be updated. Mid-level standards should also be injected about every ten injections and the end of the analysis. These standards should agree with an average percent difference ≤15% with mean obtained from the initial calibration.

8.2.4 Percent Difference (%D)

$$\%D = \frac{R1 - R2}{R1} \times 100$$

Where:
R1 = $\overline{CF}$
R2 = Calibration factor from calibration verification.

8.2.5 A Carbosorb column (or equivalent) is utilized for the quantitation analysis for all compounds except PETN (PETN is not sufficiently separated from other targets on this column).

8.2.6 A Zorbax SB-CN column (or equivalent) is utilized to confirm the analytes. If PETN is added to the target list, the Zorbax column will be utilized for the quantitation for that compound only and the Carbosorb will be used as the confirmation.

8.2.7 Retention Time Windows

Retention time (RT) windows may either be calculated (using the 72-hour study defined in SW846 method 8000B sections 7.6.1 through 7.6.4) or set as a laboratory default. For this method SWLO uses default RT window widths instead of calculating the windows for each instrument/column. The justification for using this approach on SW846 8000 series methods can be found in section 7.6 of SW846 method 8000B. "The following subsections describe one approach that may be used to establish retention time windows for GC and HPLC methods. Other approaches may be employed, provided that the analyst can demonstrate that they provide performance appropriate for the intended application."

Default RT window widths are carefully selected to minimize false negatives (windows too narrow) and false positives (windows too wide). The current default windows are listed in Table 5.

SWLO also uses the following criteria to help define the best default RT window.

1. The window width for each analyte peak has been calculated (using the 72-hour study defined in SW846 method 8000B sections 7.6.1 thru 7.6.4) and are narrower than the default window. This can be done on one instrument and used for all instruments that have identical columns and chromatographic conditions.

2. The default window width should be wider than ± ½ the width of the peak at half-height (in the high standard).

3. The default window width should be less than ± 2 times the width of the peak at half-height (in the high standard) and less than ± 2% of the absolute retention time.

Table 5
Default Retention Time Windows

CARBOSORB WINDOWS		ZORBAX SB-CN WINDOWS	
Analyte	**Time (min.)**	**Analyte**	**Time (min.)**
HMX	± 0.08	HMX	± 0.16
RDX	± 0.12	RDX	± 0.18
TNB	± 0.16	TNB	± 0.08
DNB	± 0.20	DNB	± 0.16
TETRYL	± 0.22	TETRYL	± 0.21
NB	± 0.23	NB	± 0.16
TNT	± 0.26	TNT	± 0.14
4ADNT	± 0.27	4ADNT	± 0.37
2ADNT	± 0.29	2ADNT	± 0.37
26DNT	± 0.30	26DNT	± 0.18
24DNT	± 0.31	24DNT	± 0.43
2NT	± 0.37	2NT	± 0.43
4NT	± 0.40	4NT	± 0.43
3NT	± 0.43	3NT	± 0.18
SURROGATE			
34DNT	± 0.25	34DNT	± 0.20

8.3 Sample Analysis

8.3.1 Analyze the samples using the chromatographic conditions given in section 8.1. Confirm each measurement by injecting onto the confirmation column.

8.3.2 Attachments 1-4 represent typical quantitation reports and chromatographic reports.

8.3.3 All of the soil samples are calculated based on 2.0g air-dried samples. Percent moistures are not included in the calculations.

9.0 CALCULATIONS

9.1 Calibration Factor (CF) = $\frac{\text{Peak Area (or Height) of the Compound in the Standard}}{\text{conc. in ng/mL of compound}}$

9.2 Mean CF = $\frac{\text{Sum of CF for each level}}{\text{number of levels}}$

9.3 $SD = \text{Square root}\left[\frac{\text{Sum of (CF of each individual level - Mean CF)}^2}{\text{(number of levels - 1)}}\right]$

9.4 $RSD = \frac{SD}{\text{Mean CF}} \times 100$

9.5 See SW-846, Revision 2, January 1995, page 8000B - 20 through 22 for linear and higher order fit equations.

9.6 $\% \text{ Difference} = \frac{\text{Mean CF - CF from the calibration verification standard}}{\text{Mean CF}} \times 100$

9.7 $\% \text{ Drift (used for linear and higher order fits)} = \frac{\text{Calculated conc. - Nominal conc.}}{\text{Theoretical conc.}} \times 100$

9.8 Determine the concentration of individual compounds in the sample.

9.8.1 Sample Concentration Calculation:

9.8.1.1 Aqueous Samples

Concentration (µg/L)=[As] [V(t)][D] / [Mean CF] [V(s)]

where:

As = Area (or height) of the peak for the analyte in the sample.
V(t) = Total volume of concentrated extract (mL).
D = Dilution factor.
Mean CF = Mean calibration factor (1/ng/mL)
V(s) = Volume of aqueous sample extracted (mL).

9.8.1.2 Non-aqueous Samples

Concentration (µg/Kg)=[As] [V(t)][D] / [Mean CF] [W(s)]

where:

As = Area (or height) of the peak for the analyte in the sample.
V(t) = Total volume of concentrated extract (mL).
D = Dilution factor.
Mean CF = Mean calibration factor (1/ng/mL)
W(s) = Weight of sample extracted (g).

Typist: LLR

9.9 Report results in µg/L for water samples and µg/Kg for solid samples without correction for recovery data. All QC data obtained should be reported with the sample results.

9.10 Dry weight calculation (from data gathered during step 8.1.5.1.6):

$$\% \text{ Moisture} = \frac{\text{grams of sample - grams of dried sample}}{\text{grams of sample}} \times 100$$

10.0 QUALITY ASSURANCE / QUALITY CONTROL

10.1 Prior to preparation of stock solutions, acetonitrile, methanol, and water blanks should be analyzed to determine possible interferences with analyte peaks. If the acetonitrile, methanol, or water blanks show contamination, a different batch should be used.

10.2 Method Blanks

10.2.1 Method blanks for the analysis of aqueous samples should be organic-free reagent water carried through all sample storage and handling procedures.

10.2.2 Method blanks for the analysis of soil samples should be uncontaminated soil carried through all sample storage, extraction, and handling procedures.

10.3 Laboratory Control Spikes

See "Blue Book" for current laboratory control limits.

10.4 Matrix Spikes

10.4.1 See "Blue Book" for current laboratory control limits.

10.4.2 Surrogate — To monitor extraction and analytical performance, all of the extracts are spiked with a surrogate.

11.0 WASTE DISPOSAL/POLLUTION PREVENTION

See Standard Operating Procedure "*Hazardous Waste Management Plan*," SWL-GA-114.

12.0 METHOD PERFORMANCE

Not applicable.

13.0 REFERENCES

13.1 Bauer, C.F., S.M. Koza, and T.F. Jenkins, "Collaborative Test Results for a Liquid Chromatographic Method for the Determination of Explosives Residues in Soil," manuscript submitted to the Journal of the AOAC, April 1989.

13.2 Department of the Army, "Reversed-Phase HPLC Method for the Determination of Explosive Residues in Soil," Appendix B, provided by Dennis J. Wynne, Chief,

Technology Division. U.S. Army Toxic and Hazardous Materials Agency, Aberdeen Proving Ground. Maryland 21010-5401.

13.3 Department of the Army. "An Improved RP-HPLC Method for the Determination of Nitroaromatics and Nitroamines in Water" Appendix B. provided by Dennis J. Wynne. Chief, Technology Division. U.S. Army Toxic and Hazardous Materials Agency. Aberdeen Proving Ground, Maryland 21010-5401.

14.0 DEFINITIONS

PQL:.....Practical Quantitation Limit.

N/A:.....Not applicable.

MSDS:.....Material Safety Data Sheet: Information provided by commercial vendors for the chemical products prepared by their company. The information on the MSDS includes but is not limited to composition. physical properties, hazard identification, first aid measures, . emergency clean up and/or response, exposure, and storage.

SDG: .....Sample Data Group; a unit within a single case that is used to identify a group of samples for delivery. A SDG is a group of 20 or fewer field samples within a case, received over a period of up to 14 calendar days (7 calendar day for 14-day turns.) Data from all samples in a SDG are due concurrently.

15.0 ATTACHMENTS

15.1 Attachment 1: CARBOSORB Quantitation Report and Chromatographic Report

15.2 Attachment 2: ZORBAX SB-CN Quantitation Report and Chromatographic Report

15.3 Attachment 3: CARBOSORB Quantitation and Chromatographic Report including PETN

15.4 Attachment 4: ZORBAX SB-CN Quantitation and Chromatographic Report including PETN

ATTACHMENT 1

CARBOSORB
Quantitation Report

Quantitation Report

Data File : L:\HPCHEM\LC5\DATA\5_0311\5_031102.D Vial: 2
Acq On : 11 Mar 2000 07:57 Operator: JK
Sample : L3 8-27 Inst : LC5
Misc : EXPL3 Dilution: 1.0000
IntFile : EVENTS.E
Quant Time: Mar 11 10:31 2000 Quant Results File: EXPC04.RES

Method : L:\HPCHEM\LC5\DATA\5_0311\EXPC04.M
Title-Path : - L:\HPCHEM\LC5\DATA\5_0304\
Last Update : Sun Mar 05 08:46:29 2000
Response via : Initial Calibration

Volume Inj. : 200ul
Signal Phase : CARBOSORB

Compound	R.T.	Response	ug/L	Curve
Surrogates/System Monitoring Compounds:				
7) S 3,4-DNT	13.99	112762030	754.7	A
Target Compounds:				
1) HMX	4.65	108007960	723.7	A
2) RDX	6.99	112540952	743.2	A
3) TNB	9.44	252269917	739.8	A
4) DNB	11.49	312395816	743.8	A
5) TETRYL	12.24	182479155	744.2	A
6) NB	13.07	155515668	753.1	A
8) TNT	14.76	217962709	744.7	A
9) 4ADNT	15.36	179583787	742.0	A
10) 2ADNT	16.18	212566073	744.0	A
11) 26DNT	17.22	153792017	754.6	A
12) 24DNT	17.81	259104522	734.0	A
13) 2NT	20.98	99539387	745.9	A
14) 4NT	22.40	76061145	760.5	A
15) 3NT	24.12	100800664	758.0	A

Curve Types : A=Average L=Linear P=Power Q=Quadratic/
(W)= Outside RT Window (E)= > High cal. std. (m)=manual int.

ATTACHMENT 1 (Continued)

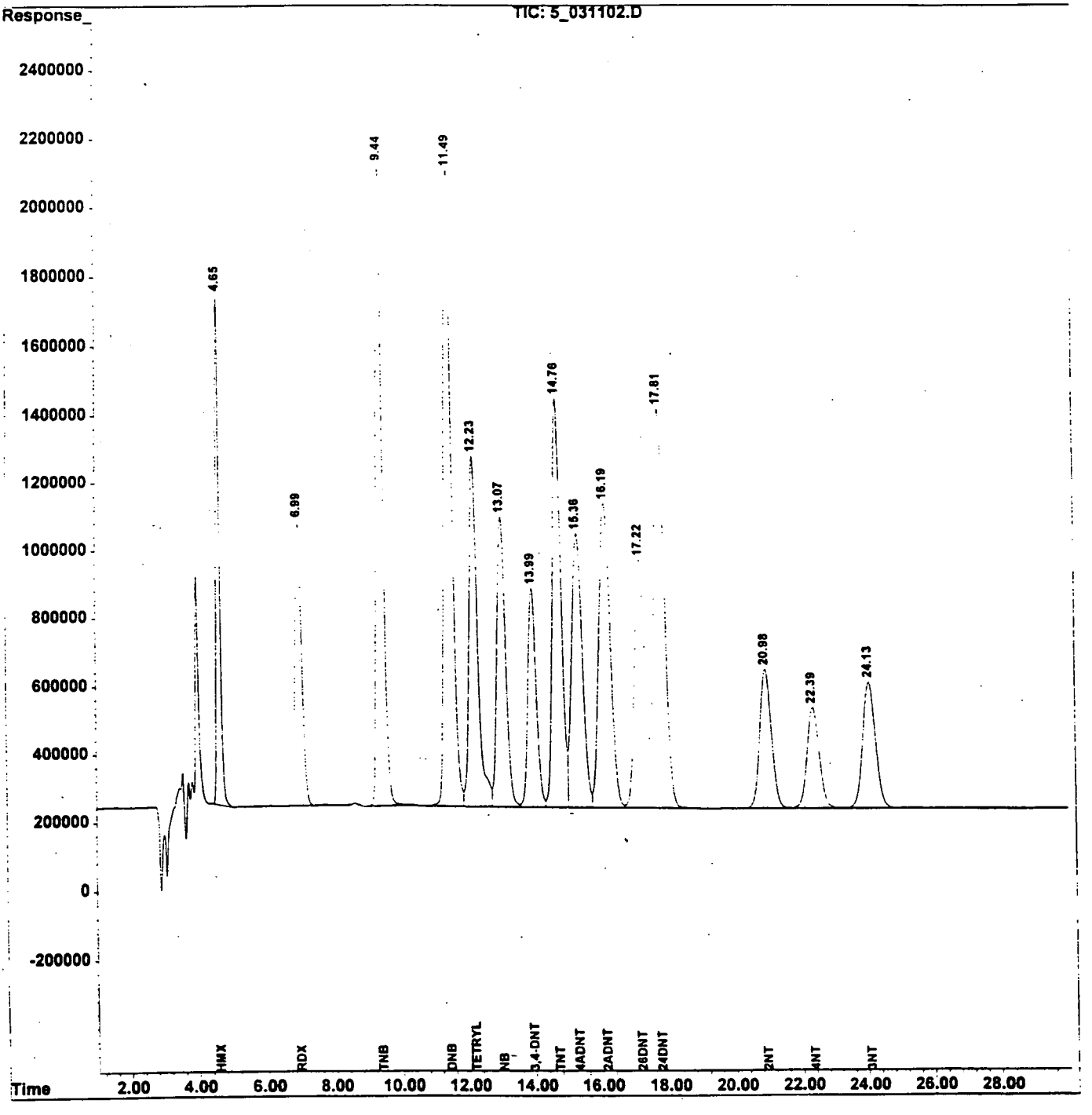
CARBOSORB
Chromatographic Report

Chromatographic Report

Data File : L:\HPCHEM\LC5\DATA\5_0311\5_031102.D Vial: 2
Acq On : 11 Mar 2000 07:57 Operator: JK
Sample : L3 8-27 Inst : LC5
Misc : EXPL3 Dilution: 1.0000
IntFile : EVENTS.E
Quant Time: Mar 11 10:31 2000 Quant Results File: EXPC04.RES

Method : L:\HPCHEM\LC5\DATA\5_0311\EXPC04.M
Title-Path : - L:\HPCHEM\LC5\DATA\5_0304\
Last Update : Sun Mar 05 08:46:29 2000
Response via : Multiple Level Calibration

Volume Inj. : 200ul
Signal Phase : CARBOSORB



ATTACHMENT 2

ZORBAX SB-CN
Quantitation Report

Quantitation Report

Data File : L:\HPCHEM\LC1\DATA\1_0311\1_031102.D
Acq On : 11 Mar 2000 07:46
Sample : L3 8-27
Misc : EXPL3
IntFile : EVENTS.E
Quant Time: Mar 13 5:28 2000 Quant Results File: CEXPC04.RES

Vial: 2
Operator: JK
Inst : LC1
Dilution: 1.0000

Method : L:\HPCHEM\LC1\DATA\1_0311\CEXPC04.M
Title-Path : - L:\HPCHEM\LC1\DATA\1_0304\
Last Update : Tue Mar 07 09:21:08 2000
Response via : Initial Calibration

Volume Inj. : 200ul
Signal Phase : ZORBAX SB-CN

Compound	R.T.	Response	ug/L	Curve
Surrogates/System Monitoring Compounds:				
7) S 3,4-DNT	13.39	47807892	793.2	A
Target Compounds:				
1) HMX	8.06	31578571	723.0	A
2) RDX	7.56	40376951	741.3	A
3) TNB	5.75	89334822	793.3	A
4) DNB	6.65	196055633	789.8	A
5) TETRYL	15.34	60277211	808.3	A
6) NB	6.65	196055633	789.8	A
8) TNT	9.72	84846307	785.8	A
9) 4ADNT	14.32	142031486	789.0	A
10) 2ADNT	14.32	142031486	789.0	A
11) 26DNT	11.16	113779264	817.4	A
12) 24DNT	10.65	194882858	798.9	A
13) 2NT	10.65	194882858	798.9	A
14) 4NT	10.65	194882858	798.9	A
15) 3NT	11.16	113779264	817.4	A

Curve Types : A=Average L=Linear P=Power Q=Quadratic/
(W)= Outside RT Window (E)= > High cal. std. (m)=manual int.

ATTACHMENT 2 (Continued)

ZORBAX SB-CN
Chromatographic Report

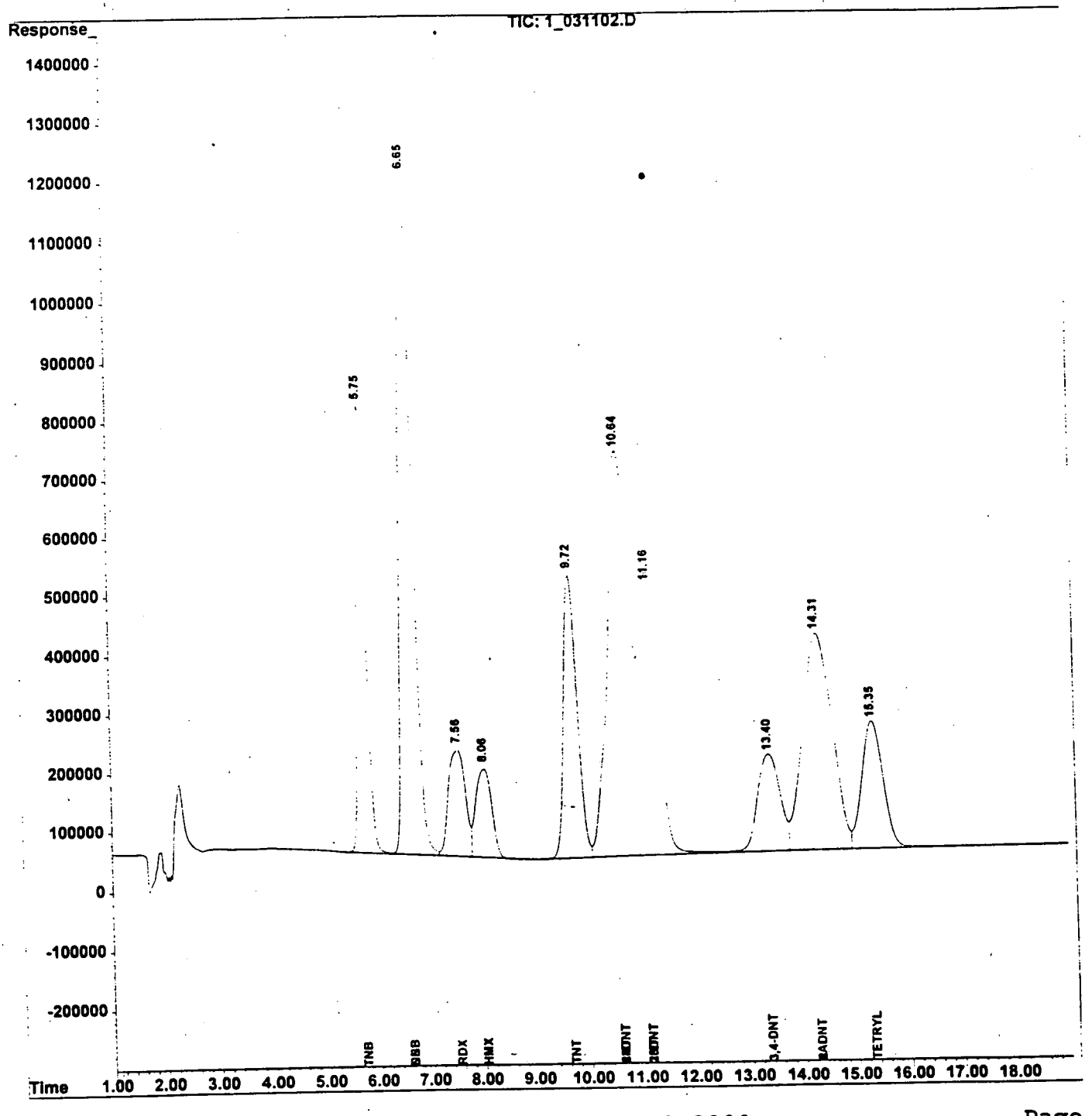
Chromatographic Report

Data File : L:\HPCHEM\LC1\DATA\1_0311\1_031102.D
Acq On : 11 Mar 2000 07:46
Sample : L3 8-27
Misc : EXPL3
IntFile : EVENTS.E
Quant Time: Mar 13 5:28 2000 Quant Results File: CEXPC04.RES

Vial: 2
Operator: JK
Inst : LC1
Dilution: 1.0000

Method : L:\HPCHEM\LC1\DATA\1_0311\CEXPC04.M
Title-Path : - L:\HPCHEM\LC1\DATA\1_0304\
Last Update : Tue Mar 07 09:21:08 2000
Response via : Multiple Level Calibration

Volume Inj. : 200ul
Signal Phase : ZORBAX SB-CN



ATTACHMENT 3

CARBOSORB
Quantitation Report including PETN

Quantitation Report

Data File : L:\HPCHEM\LC1\DATA\1_0217\1_021732.D
Acq On : 18 Feb 2000 02:24
Sample : L3 8-48
Misc : PETNL3
IntFile : EVENTS.E
Quant Time: Feb 18 16:32 2000 Quant Results File: PETNB17.RES

Vial: 32
Operator: JK
Inst : LC1
Dilution: 1.0000

Method : L:\HPCHEM\LC1\DATA\1_0217\PETNB17.M
Title-Path : - L:\HPCHEM\LC1\DATA\1_0217\
Last Update : Fri Feb 18 16:28:15 2000
Response via : Initial Calibration

Volume Inj. : 200ul
Signal Phase : CARBOSORB

Compound	R.T.	Response	ug/L	Curve
Surrogates/System Monitoring Compounds:				
7) S 3,4-DNT	16.02	81700478	811.8	A
Target Compounds:				
1) HMX	4.80	54964424	795.8	A
2) RDX	7.39	67652353	858.9	A
3) TNB	10.15	154512182	817.0	A
4) DNB	12.51	215241172	822.8	A
5) TETRYL	13.69	113188860	839.3	A
6) NB	14.47	143853424	826.2	A
8) TNT	16.51	151894233	812.9	A
9) 4ADNT	17.33	102171686	813.7	A
10) 2ADNT	18.12	147527368	812.2	A
11) 26DNT	19.55	96673303	810.8	A
12) 24DNT	20.12	197867722	811.3	A
13) 2NT	24.20	177124221	822.9	A
14) 4NT	25.89	139133115	869.9	A
15) 3NT	27.97	219327218	823.8	A
16) PETN	28.71	76315216	818.0	A

Curve Types : A=Average L=Linear P=Power Q=Quadratic/
(W)= Outside RT Window (E)= > High cal. std. (m)=manual int.

ATTACHMENT 3 (Continued)

CARBOSORB
Chromatographic Report including PETN

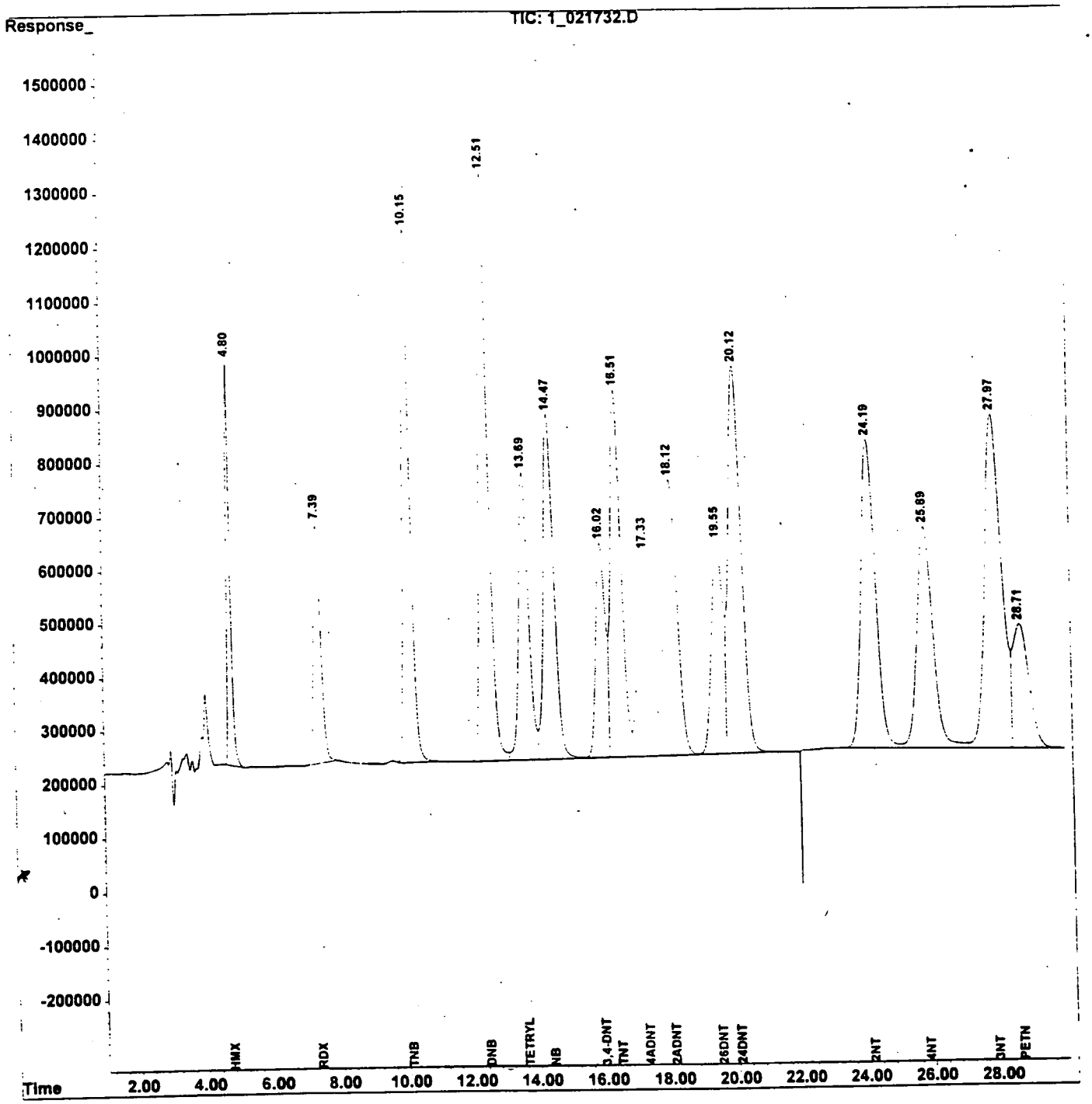
Chromatographic Report

Data File : L:\HPCHEM\LC1\DATA\1_0217\1_021732.D
Acq On : 18 Feb 2000 02:24
Sample : L3 8-48
Misc : PETNL3
IntFile : EVENTS.E
Quant Time: Feb 18 16:32 2000 Quant Results File: PETNB17.RES

Vial: 32
Operator: JK
Inst : LC1
Dilution: 1.0000

Method : L:\HPCHEM\LC1\DATA\1_0217\PETNB17.M
Title-Path : - L:\HPCHEM\LC1\DATA\1_0217\
Last Update : Fri Feb 18 16:28:15 2000
Response via : Multiple Level Calibration

Volume Inj. : 200ul
Signal Phase : CARBOSORB



ATTACHMENT 4

ZORBAX SB-CN
Quantitation Report including PETN

Quantitation Report

Data File : L:\HPCHEM\LC5\DATA\5_0214\5_021404.D Vial: 4
Acq On : 14 Feb 2000 15:53 Operator: JK
Sample : L3 5-48-8 Inst : LC5
Misc : PETNL3 Dilution: 1.00
IntFile : EVENTS.E
Quant Time: Jul 12 10:49 2000 Quant Results File: CPETNB14.RES

Method : L:\HPCHEM\LC5\DATA\5_0214\CPETNB14.M
Title-Path : - L:\HPCHEM\LC5\DATA\5_0213\
Last Update : Wed Jul 12 10:47:20 2000
Response via : Initial Calibration

Volume Inj. : 200ul
Signal Phase : ZORBAX SB-CN
Signal Info :

Compound	R.T.	Response	ug/L	Curve
System Monitoring Compounds:				
7) S 3,4-DNT	13.00	64847378	759.9	A
Target Compounds:				
1) HMX	8.09	58186565	727.8	A
2) RDX	7.60	61129203	709.2	A
3) TNB	5.78	134387535	726.2	A
4) DNB	6.63	235250174	721.4	A
) TETRYL	15.15	88123970	715.9	A
NB	6.63	235250174	721.4	A
8) TNT	9.61	115971884	729.5	A
9) 4ADNT	13.88	200976416	723.4	A
10) 2ADNT	13.88	200976416	723.4	A
11) 26DNT	10.92	129977390	717.6	A
12) 24DNT	10.50	223321600	739.4	A
13) 2NT	10.50	223321600	739.4	A
14) 4NT	10.50	223321600	739.4	A
15) 3NT	10.92	129977390	717.6	A
16) PETN	25.31	73002910	800.0	A

Curve Types : A=Average L=Linear P=Power Q=Quadratic/
(f)=RT Delta > 1/2 Window (E)= > High cal. std. (m)=manual int.

ATTACHMENT 4 (Continued)

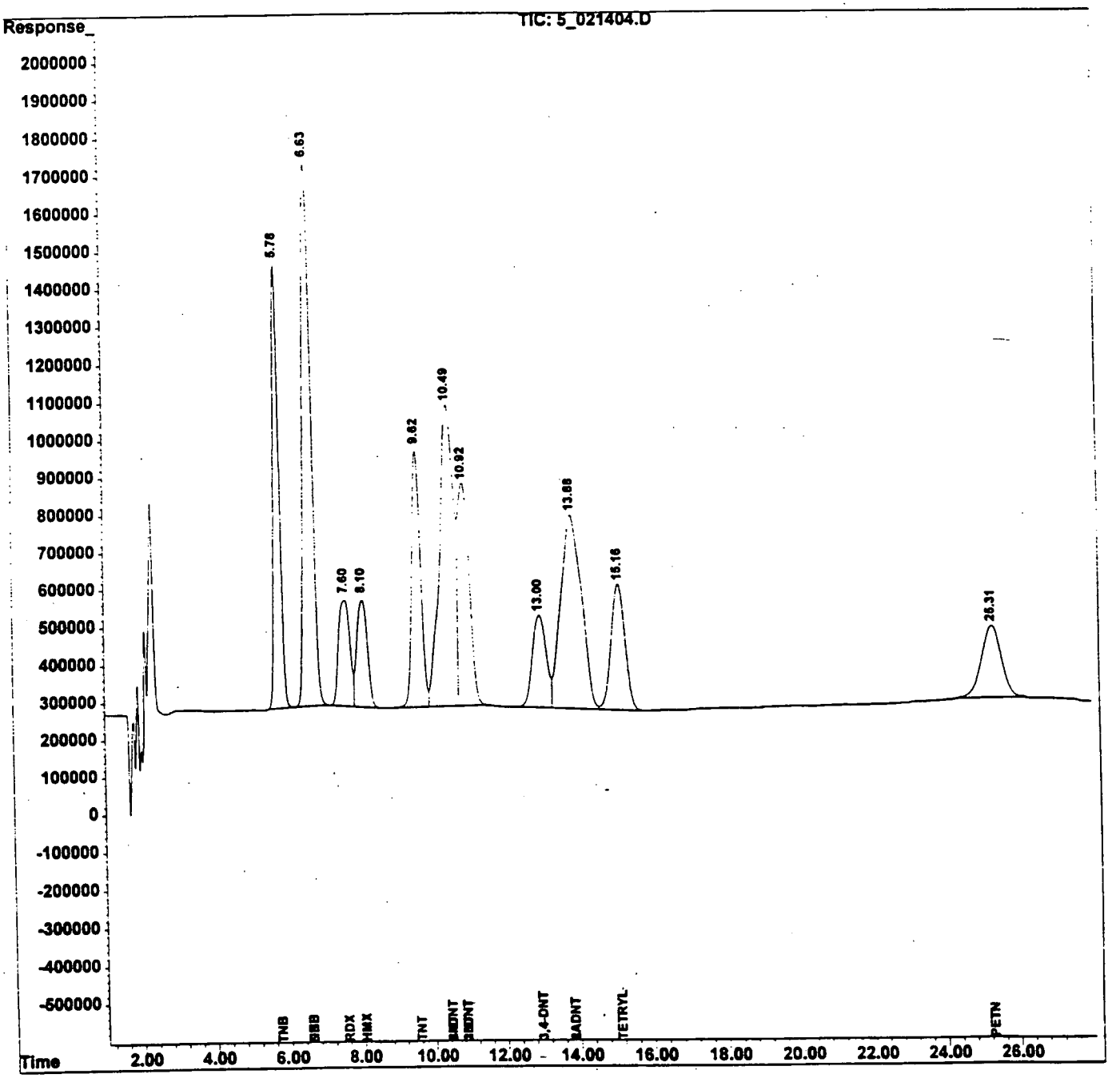
ZORBAX SB-CN
Chromatographic Report including PETN

Chromatographic Report

Data File : L:\HPCHEM\LC5\DATA\5_0214\5_021404.D Vial: 4
Acq On : 14 Feb 2000 15:53 Operator: JK
Sample : L3 5-48-8 Inst : LC5
Misc : PETNL3 Dilution: 1.00
IntFile : EVENTS.E
Quant Time: Jul 12 10:49 2000 Quant Results File: CPETNB14.RES

Method : L:\HPCHEM\LC5\DATA\5_0214\CPETNB14.M
Title-Path : - L:\HPCHEM\LC5\DATA\5_0213\
Last Update : Wed Jul 12 10:47:20 2000
Response via : Multiple Level Calibration

Volume Inj. : 200ul
Signal Phase : ZORBAX SB-CN
Signal Info :



SOUTHWEST LABORATORY OF OKLAHOMA, INC. and
AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.

TM

Standard Operating Procedure

Polynuclear Aromatic Hydrocarbons (PAH) by HPLC

Document No.: SWL-OL-201

Rev No. / Date: Rev. 3.2 — 07/27/00

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APPROVALS	DATES
Procedure Prepared By	07/27/00 Date
Program Manager or Section Supervisor	7-27-00 Date
Laboratory Safety Officer	7/27/2000 Date
Laboratory QA/QC Officer	7/28/2000 Date

(Effective Date is 5 calendar days after the last signature above - QA/QC Officer)

Document Status

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POLYNUCLEAR AROMATIC HYDROCARBONS (PAH) BY HPLC

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1.0 SCOPE AND APPLICATION

1.1 EPA Method 8310 is used to determine the concentrations of certain polynuclear aromatic hydrocarbons (PAH) in ground water and wastes. EPA Method 610 may also be used to determine PAHs in water samples provided that it is modified to use the calibration procedures that are listed in Section 8.2. Specifically, these methods are used to determine the following:

TABLE 1 - COMPOUND LIST

Analytes			
Naphthalene	91-20-3	Benzo(a)anthracene	56-55-3
Acenaphthylene	208-96-8	Chrysene	218-01-9
Acenaphthene	83-32-9	Benzo(b)fluoranthene	205-99-2
Fluorene	86-73-7	Benzo(k)fluoranthene	207-08-9
Phenanthrene	85-01-8	Benzo(a)pyrene	50-32-8
Anthracene	120-12-7	Dibenzo(a,h)anthracene	53-70-3
Fluoranthene	206-44-0	Benzo(ghi)perylene	191-24-2
Pyrene	129-00-0	Indeno(1,2,3-cd)pyrene	193-39-5
Surrogates			
2-Fluorobiphenyl	321-60-8	p-Terphenyl	92-94-4

1.2 A limitation of this analysis is the high possibility of identifying false hits as targets. Use of Method 8310 presupposes a high expectation of finding the specific compounds of interest. Method 610 suggests that all unfamiliar samples be confirmed by GC/MS analysis (EPA Method 625.) It is the policy of this laboratory that the analysis of PAHs will be reported without GC/MS confirmation unless it is specifically requested by the client.

1.3 The practical quantitation limits (PQL's) are listed in Table 2. The sensitivity of this method usually depends on the level of interferences rather than instrumental limitations. When interferences are present, the level of sensitivity will be lower. The PQL's are based on the low point (L1) in the initial calibration represented in Table 3, the amount of sample extracted, and the volume of the extract.

1.4 This method is recommended for use only by experienced HPLC analysts or under the close supervision of such qualified persons.

2.0 METHOD SUMMARY

2.1 Method 8310 provides high performance liquid chromatographic (HPLC) conditions for the detection of ppb levels of certain polynuclear aromatic hydrocarbons. Two detectors are utilized to analyze the compounds. A UV detector and a Fluorescence detector are connected in series for confirmation of target analytes.

2.2 All extracts, including laboratory QC, undergo silica gel column cleanup (Method 3630).

TABLE 2 - Practical Quantitation Limits

Compound	Water (ug/L)	Soil (ug/Kg)
Naphthalene	0.2	10
Acenaphthylene	0.2	10
Acenaphthene	0.2	10
Fluorene	0.2	10
Phenanthrene	0.2	10
Anthracene	0.2	10
Fluoranthene	0.2	10
Pyrene	0.2	10
Benzo(a)Anthracene	0.2	10
Chrysene	0.2	10
Benzo(b)Fluoranthene	0.2	10
Benzo(k)Fluoranthene	0.2	10
Benzo(a)Pyrene	0.2	10
Dibenzo(a,h)Anthracene	0.2	10
Benzo(g,h,i)Perylene	0.2	10
Indeno-(1,2,3-c,d)Pyrene	0.2	10

TABLE 3 - Calibration Standards (µg/L)

Compound	L1	L2	L3	L4	L5
Naphthalene	0.1	0.2	0.4	0.8	1.6
Acenaphthylene	0.1	0.2	0.4	0.8	1.6
Acenaphthene	0.1	0.2	0.4	0.8	1.6
Fluorene	0.1	0.2	0.4	0.8	1.6
Phenanthrene	0.1	0.2	0.4	0.8	1.6
Anthracene	0.1	0.2	0.4	0.8	1.6
Fluoranthene	0.1	0.2	0.4	0.8	1.6
Pyrene	0.1	0.2	0.4	0.8	1.6
Benzo(a)Anthracene	0.1	0.2	0.4	0.8	1.6
Chrysene	0.1	0.2	0.4	0.8	1.6
Benzo(b)Fluoranthene	0.1	0.2	0.4	0.8	1.6
Benzo(k)Fluoranthene	0.1	0.2	0.4	0.8	1.6
Benzo(a)Pyrene	0.1	0.2	0.4	0.8	1.6
Dibenzo(a,h)Anthracene	0.1	0.2	0.4	0.8	1.6
Benzo(g,h,i)Perylene	0.1	0.2	0.4	0.8	1.6
Indeno-(1,2,3-c,d)Pyrene	0.1	0.2	0.4	0.8	1.6

3.0 HEALTH AND SAFETY

Standard precautionary measures used for handling other organic compounds should be sufficient for safe handling of the analytes targeted by method 8310.

4.0 SAMPLE PRESERVATOIN, CONTAINERS, HANDLING & STORAGE

Extraction of aqueous samples must be started within 5 days of sample receipt and soil samples within 10 days of receipt. Extracts are stored until analysis at 4°C and must be analyzed within 40 days of extraction.

5.0 INTERFERENCES

5.1 Solvents, 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 under the conditions of the analysis by analyzing method blanks. Specific selection of reagents and purification of solvents by distillation in all glass systems may be required.

5.2 Interferences co-extracted from the samples will vary considerably from source to source. Although a general cleanup technique is provided as part of this method, individual samples may require additional cleanup approaches to achieve the level of detection stated in Table 2.

5.3 The chromatographic conditions described allow for a unique resolution of the specific PAH compounds covered by this method. Other PAH compounds, in addition to matrix artifacts, may interfere.

6.0 HPLC APPARATUS

6.1 Autosampler

6.2 Pumps: 2 high pressure pumps capable of 6000 psi at 10.0 mL/min

6.3 Gradient Pump Controller

6.4 Column: Supelco LC-PAH 5µ, 10cm X 4.6mm ID column, or equivalent

6.5 Column Heater

6.6 Detectors: 1) Variable Wavelength UV detector set to 254 nm. 2) Variable excitation/ Filter emission Fluorescence detector with the excitation wavelength set to 250 nm and equipped with a 320nm (or equivalent) cut off emission filter.

6.7 Data System - Hewlett Packard Enviroquant

7.0 REAGENTS

7.1 HPLC grade chemicals shall be used in all tests. It is intended that all reagents shall conform to the specification of the committee on analytical reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficient high purity to permit its use without lowering the accuracy of the determination.

7.1.1 Acetonitrile. CH3CN - HPLC grade.

7.1.2 Water, H2O - HPLC grade

7.2 Stock standard solutions — Certified standard mixes obtained from a vendor that contains the target analytes.

7.3 Working Calibration standards — Prepare 5 working calibration standards by diluting the stock standards with 50:50 (v/v) with a 5 g/L (0.5%) Calcium Chloride/water and acetonitrile. The low standard is at the PQL.

7.4 LCS (Laboratory Control Spike) and LCSD (Laboratory Control Spike Duplicate) — LCS's are spiked with the analytes of interest at the midpoint level of the calibration curve.

7.5 MS (Matrix Spike) and MSD (Matrix Spike Duplicate) — MS and MSD are spiked with the analytes of interest at the midpoint level of the calibration curve.

7.6 Surrogates — Due to matrix interferences that are often present in the analysis, two surrogates are used. 2F-biphenyl and p-terphenyl are the analytes of non-interest used for the surrogates. Both surrogates are spiked at the midpoint level of the calibration curve.

7.7 Eluent — To prepare 1 liter of 50% acetonitrile/water, add 500 mL of acetonitrile to 500 mL of Millipore or organic free water.

8.0 PROCEDURE

8.1 Chromatographic Conditions

Flow Rate:	1.4 mL/min
Injection Volume:	50 µL
Column Temperature:	35° C
Run Time:	30 minutes
Solvent Gradient:	Initial - 45% / 55% Acetonitrile / Water
	2-16 minutes - Linear ramp up to 100% Acetonitrile
	21-23 minutes - Linear ramp back to initial conditions

8.2 Calibration of HPLC

8.2.1 Following SW-846, Method 8310, a five point initial calibration is used. The average %RSD of the calibration factors should be ≤20%. Immediately following

the injection of the initial calibration, an initial calibration verification (ICV), obtained from a different vendor than the initial calibration standards, is injected.

The concentration of the ICV is diluted to equal the concentration of the mid level standard.

8.2.2 Calibration factor (CF):

$CF = \frac{\text{Peak Area of Standard}}{\text{Concentration (ug/L)}}$ ①	$\%RSD = \frac{STD \times 100}{\overline{CF}}$ ③
$\overline{CF} = \sum_{i=1}^{n} \frac{CF}{n}$ ②	Where: $\overline{CF}$ = Mean of the calibration factors N = Total number of calibration level STD = Standard deviation of the calibration factors CF = Calibration factors

8.2.3 A calibration verification using the mid level standard should be injected at the beginning of an analysis day and the retention times should be updated. Mid level standards should also be injected about every ten injections and at the end of the analysis. These standards should agree with an average percent difference ≤ 15% with mean obtained from the initial calibration. If a continuing calibration fails, it may be reinjected and analyses restarted from that point if it meets specifications on reinjection. If it fails again, the initial calibration must be repeated.

8.2.4 Percent Difference (%D)

$$\%D = \frac{R1 - R2}{R1} \times 100$$

Where:
R1 = $\overline{CV}$
R2 = Calibration factor from the calibration verification.

8.2.5 Retention Time Windows
Retention time (RT) windows may either be calculated (using the 72-hour study defined in SW846 method 8000B sections 7.6.1 through 7.6.4) or set as a laboratory default. For this method SWLO uses default RT window widths instead of calculating the windows for each instrument/column. The justification for using this approach on SW846 8000 series methods can be found in section

7.6 of SW846 method 8000B. "The following subsections describe one approach that may be used to establish retention time windows for GC and HPLC methods. Other approaches may be employed, provided that the analyst can demonstrate that they provide performance appropriate for the intended application."

Default RT window widths are carefully selected to minimize false negatives (windows too narrow) and false positives (windows too wide). The current default windows are listed in Table 4.

SWLO also uses the following criteria to help define the best default RT window.

1. The window width for each analyte peak has been calculated (using the 72-hour study defined in SW846 method 8000B sections 7.6.1 thru 7.6.4) and are narrower than the default window. This can be done on one instrument and used for all instruments that have identical columns and chromatographic conditions.

2. The default window width should be wider than ± ½ the width of the peak at half-height (in the high standard).

The default window width should be less than ± 2 times the width of the peak at half-height (in the high standard) and less than ± 2% of the absolute retention time.

Table 4
Default Retention Time Windows

Compound	Time (min)
Naphthalene	± 0.09
Acenaphthylene	± 0.09
Acenaphthene	± 0.09
Fluorene	± 0.14
Phenanthrene	± 0.14
Anthracene	± 0.14
Fluoranthene	± 0.14
Pyrene	± 0.14
Benzo(a)Anthracene	± 0.18
Chrysene	± 0.23
Benzo(b)Fluoranthene	± 0.23
Benzo(k)Fluoranthene	± 0.23
Benzo(a)Pyrene	± 0.23
Dibenzo(a,h)Anthracene	± 0.27
Benzo(g,h,i)Perylene	± 0.32
Indeno-(1,2,3-c,d)Pyrene	± 0.36
Surrogates	
2F-Biphenyl	± 0.09
p-Terphenyl	± 0.18

8.3 Sample analysis

8.3.1 Analyze the samples using the chromatographic conditions given in section 8.1.

8.3.2 The fluorescent detector is used for quantitation and the UV detector is used for the confirmation of all compounds except for acenaphthylene, which does not fluoresce. If acenaphthalene is present in a sample, it will be reported from the UV detector without confirmation. Refer to Attachments 1 and 2 for a sample quantitation report and chromatographic report.

8.3.3 Calculation for positive hits:

$$\text{Concentration } (\mu g/L \text{ or } \mu g/Kg) = \frac{(A)(Vt)(DF)}{(\overline{CF})(Vo)}$$

Where:

A	=	Area of the peak for the compound to be measured
$\overline{CF}$	=	Mean calibration factor (1/(µg/L))
Vo	=	Volume of water (L) or amount of soil extracted (Kg)
Vt	=	Volume of the concentrate extracted (mL)
DF	=	Dilution factor

9.0 CALCULATIONS

See formulas list in Sections 8.2 and 8.3 above.

10.0 QUALITY ASSURANCE/QUALITY CONTROL

10.1 Prior to preparation of stock solutions, acetonitrile and water blanks should be analyzed to determine possible interferences with analyte peaks. If the acetonitrile and water blanks show contamination, a different batch should be used.

10.2 Method blank

10.2.1 Method blanks for the analysis of aqueous samples should be organic-free reagent water carried through all sample storage and handling procedures.

10.2.2 Method blanks for the analysis of soil samples should be uncontaminated soil carried through all sample storage, extraction, and handling procedures.

10.3 Laboratory Control Spike (LC) and Laboratory Control Spike Duplicate (LD)
See "Blue Book" for current laboratory control limits.

10.4 Matrix Spike (MS) and Matrix Spike Duplicate (MSD)
See "Blue Book" for current laboratory control limits.

10.5 Surrogates

10.5.1 To monitor extraction and analytical performance, all of the extracts are spiked with two surrogates.

10.5.2 If one of the surrogates is within control limits the sample is not re-extracted.

11.0 WASTE DISPOSAL/POLLUTION PREVENTION

See Standard Operating Procedure "Hazardous Waste Management Plan", SWL-GA-114.

12.0 METHOD PERFORMANCE

See Performance Criteria cited in Section 8.2.

13.0 REFERENCES

13.1 United States Environmental Protection Agency, "Determination of Polycyclic Aromatic Hydrocarbons in Ground Water and Wastes," SW846 Method 8310, 1986.

13.2 United States Environmental Protection Agency, "Determinative Chromatographic Separations," SW846 Method 8300B, Revision 2, 1996.

13.3 United States Environmental Protection Agency, "Polynuclear Aromatic Hydrocarbons," SW-846 Method 610, from 7-1-99 edition of 40 CFR Ch. 1.

14.0 DEFINITIONS

14.1 PAH.....Polynuclear aromatic hydrocarbon (PAH)

14.2 HPLC.....High performance liquid chromatograph

14.3 UV Detector.....Ultraviolet detector

14.4 CF.....Calibration factor — A numeric factor that is determined by dividing the area counts of a compounds' response by the amount injected in (ng).

14.5 PQL.....The Practical Quantitation Limit

15.0 ATTACHMENTS

15.1 Attachment 1: Quantitation Report

15.2 Attachment 2: Chromatographic Report

ATTACHMENT 1
QUANTITATION REPORT

Quantitation Report

Signal #1 : L:\HPCHEM\LC2\DATA\2_0305\2_030511.D\data.ms Vial: 11
Signal #2 : L:\HPCHEM\LC2\DATA\2_0305\2_030511.D\CONFIRM.D\data.ms
Acq On : 05 Mar 2000 22:48 Operator: JB
Sample : L3 8-57 Inst : LC2
Misc : PAHL3 Dilution: 1.0000
IntFile Signal #1: EVENTS.E IntFile Signal #2: EVENTS2.E
Quant Time: Mar 6 8:28 2000 Quant Results File: PAHB24.RES

Method : L:\HPCHEM\LC2\DATA\2_0305\PAHB24.M
Title-Path : - L:\HPCHEM\LC2\DATA\2_0224\
Last Update : Thu Feb 24 23:05:09 2000
Response via : Initial Calibration

Volume Inj. : 50ul
Signal Phase : LC-PAH

	Compound	RT#1	RT#2	Resp#1	Resp#2	ug/L	ug/L
	Surrogates/System Monitoring Compounds:						
3)	2-F BIPHENYL	6.45	6.56	159.4E6	154.9E6	1533.0	1417.4
10)	TERPHENYL	14.02	14.13	28547169	419.5E6	394.7	392.4
	Target Compounds:						
1)	NAPHTHALENE	4.96	5.08	11678778	17224649	406.6	317.9
2)	ACENAPHTHYLENE	5.96	0.00	7895728	0	390.2	N.D. #
4)	ACENAPHTHENE	7.50	7.61	3943720	169.5E6	375.0	364.4
5)	FLUORENE	7.96	8.07	54525912	119.0E6	398.8	361.1
	PHENANTHRENE	9.04	9.15	179.3E6	109.3E6	386.6	352.6
	ANTHRACENE	10.20	10.32	401.2E6	837.4E6	389.9	389[illegible]
	FLUORANTHENE	11.17	11.29	40321240	212.8E6	387.9	370.[illegible]
9)	PYRENE	12.01	12.13	29797908	79991917	382.7	346.6
11)	BENZO (a) ANTHRACENE	14.69	14.81	85246254	151.5E6	396.2	367.1
12)	CHRYSENE	15.44	15.55	115.9E6	100.4E6	404.6	364.1
13)	BENZO (b) FLUORANTHEN	17.25	17.36	98389023	421.2E6	390.8	386.9
14)	BENZO (k) FLUORANTHEN	18.24	18.35	88552114	1259.8E6	379.3	379.0
15)	BENZO (a) PYRENE	18.96	19.08	78791915	210.8E6	408.7	387.8
16)	DIBENZO (a,h) ANTHRAC	20.01	20.12	19498083	42794112	407.3	345.1
17)	BENZO (g,h,i) PERYLEN	20.76	20.88	34374052	48689095	409.8	343.7
18)	INDENO (1,2,3-cd) PYR	21.48	21.59	108.5E6	142.3E6	385.4	356.7

(W)= Outside RT Window (#)=Amounts differ by > 40% (m)=manual int.
(E)= > Highest cal. std. (d)=compound deleted (H) Dil. < med. std

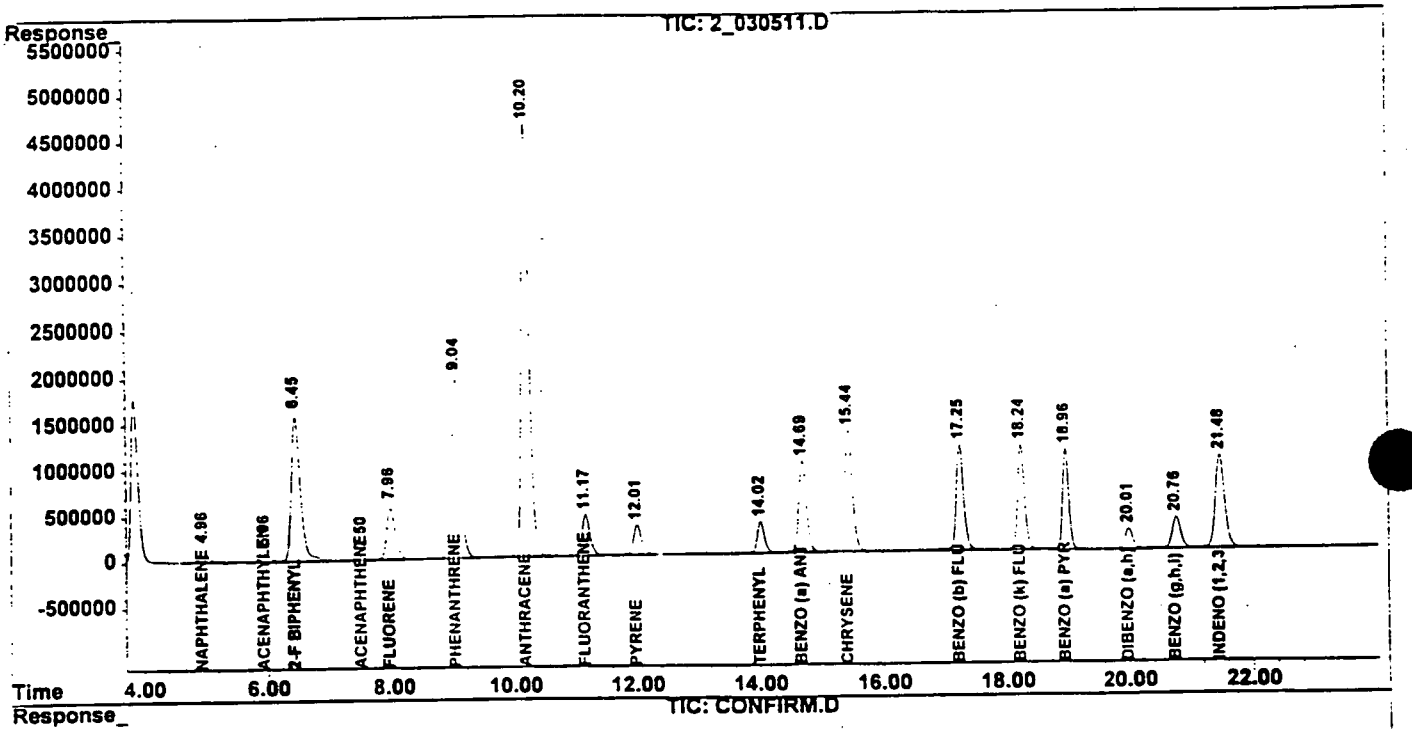
ATTACHMENT 2
CHROMATOGRAPHIC REPORT

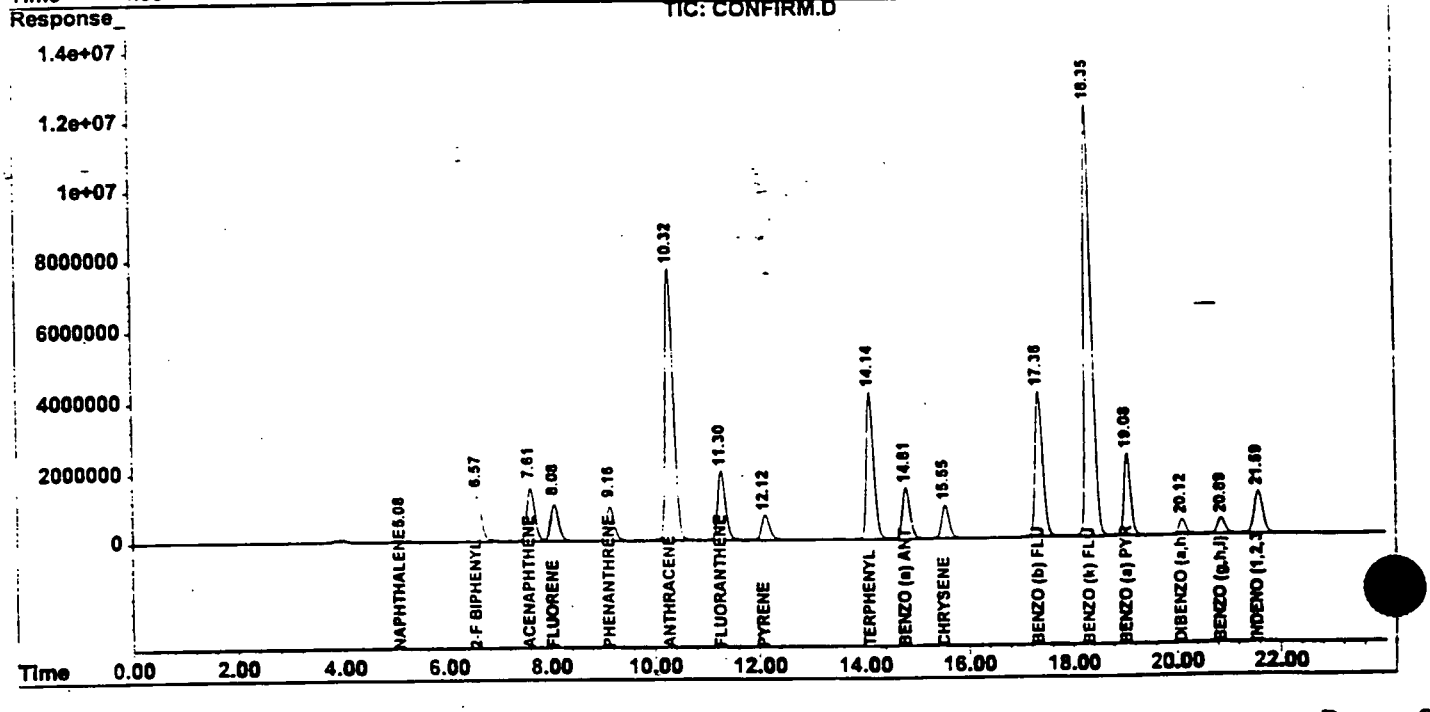
Chromatographic Report

Signal #1 : L:\HPCHEM\LC2\DATA\2_0305\2_030511.D\data.ms Vial: 11
Signal #2 : L:\HPCHEM\LC2\DATA\2_0305\2_030511.D\CONFIRM.D\data.ms
Acq On : 05 Mar 2000 22:48 Operator: JB
Sample : L3 8-57 Inst : LC2
Misc : PAHL3 Dilution: 1.000
IntFile Signal #1: EVENTS.E IntFile Signal #2: EVENTS2.E
Quant Time: Mar 6 8:28 2000 Quant Results File: PAHB24.RES

Method : L:\HPCHEM\LC2\DATA\2_0305\PAHB24.M
Title-Path : - L:\HPCHEM\LC2\DATA\2_0224\
Last Update : Thu Feb 24 23:05:09 2000
Response via : Multiple Level Calibration

Volume Inj. : 50ul
Signal Phase : LC-PAH





SOUTHWEST LABORATORY OF OKLAHOMA, INC. *and*
AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.

TM

Standard Operating Procedure

Preventative Maintenance of Gas Chromatographs

Document No.: SWL-OP-201

Rev No. / Date: Rev. 1.5 — 06/09/99

APPROVALS	DATES
Procedure Prepared By	Date
Program Manager or Section Supervisor	Date
Laboratory Safety Officer	Date
Laboratory QA/QC Officer	Date

(Effective Date is 5 calendar days after the last signature above - QA/QC Officer)

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PREVENTATIVE MAINTENANCE OF GAS CHROMATOGRAPHS

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1.0 SCOPE AND APPLICATION. This document provides guidance for the scheduling, performance, and documentation of routine maintenance for Hewlett-Packard gas chromatographs.

2.0 METHOD SUMMARY

2.1 Preventative maintenance is performed as scheduled in this document or on an as needed basis if evaluation reveals that preventative maintenance is not necessary.

2.2 After every maintenance, all work performed will be documented in the "instrument maintenance log" specific to that instrument.

2.2.1 Document: Problems noted, maintenance performed, H_2 instruments were thoroughly leak tested, date of maintenance, date of return to analytical control, and initials of the one performing maintenance.

3.0 HEALTH AND SAFETY

3.1 See Standard Operating Procedure, "Laboratory Safety Plan", SWL-GA-111.

3.2 Activated charcoal traps should be installed on every GC effluent that may vent sample extract.

3.3 Rinse vials must be capped and changed daily.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING & STORAGE. N/A

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

5.1 Contaminated gas will severely hinder chromatographic analyses, and may severely damage chromatographic systems. Contaminated gas may cause: 1) baseline signal rise, 2) peak tailing, and/or 3) column degradation. Certain contaminants may adhere to GC lines and valves after the source of contamination has been removed, thus necessitating extensive maintenance. Oxygen will severely reduce the life of analytical columns. Always cool the instrument and detector before opening the closed chromatographic system to air.

5.2 Oxygen/moisture indicator traps must be installed for each separate source of gas (cylinder or generator). The traps must be checked daily to ensure that contaminated gas does not reach the instrumentation.

5.3 GC problems that are unable to be remedied in-house, and/or require non-routine or expensive parts, will be deferred to Hewlett-Packard Service. The telephone number is 1-800-424-9759. Have the instrument model and serial number ready when calling.

NOTE: Before calling HP Service, the Section Supervisor or Manager must be notified.

6.0 EQUIPMENT/APPARATUS

6.1 Replacement parts including but not limited to, syringes, septum, inlet liners, inlet seals (gold seals), column nuts, weldmet assemblies, split/purge valves, needle valves, mass flow controllers, various ferrules, columns, pencil traps, ECDs, heating elements, cooling and oven fans, and integrated circuit boards.

6.2 Tools and apparatus may include but are not limited to, metric and standard wrenches, column cutters, dental picks, Philips and flat head screw-drivers, flow meters, leak detectors, bubble meters (not to be used for H_2), and other instrument-specific tools.

7.0 REAGENTS

7.1 Methylene chloride, methanol, hexane – pesticide or reagent grade or equivalent.

7.2 Activated charcoal

8.0 PROCEDURE

8.1 Tables 1 and 2 present a schedule for preventative maintenance/part replacement for all Hewlett-Packard Gas Chromatographs. The schedules will be followed unless instrument evaluation reveals that a particular maintenance is unnecessary.

TABLE 1
Gas Chromatograph Preventative Maintenance/Replacement Schedule

Item	Replacement Time	Troubleshooting / Comments
Activated Charcoal Trap	Every month or sooner	Traps potentially dangerous compounds after they have passed through the GC detectors and split vents.
Column	As needed	A new column is required for pesticide/PCB analyses if: resolution falls below 60%, if breakdown (not caused by other problems) cannot be removed by cutting off the front portion of the guard column, or any other problem traced to the column. Indicators of the end of a column's life for other analysis types include: peak tailing, baseline rise, loss of sensitivity, retention time instability, loss of linearity and loss of calibration verification reproducibility.
ECD	As needed	Refurbishing is recommended when the detector signal rises 100% above the original signal or the baseline will not stabilize. Baking the detectors with hydrogen and/or cleaning the detector anode may make refurbishing unnecessary.
NPD Bead	As needed	Refurbishing/replacing of the bead is recommended when the detector loses stability (high drift) or when severe peak tailing precludes sound analyses. See refurbishing kit instructions for the procedure.
Detector Nut Ferrule	3 months or as needed	Retention time shifts may be indicative of a faulty or old detector nut ferrule.
Injection Liner (& O-ring)	2 weeks or as needed	To ensure contaminant-free chromatography and to prevent analyte breakdown. Petroleum analyses using a FID do not require liner changes as frequently as for other analyses.
Injection Port	As needed	To ensure contamination-free chromatography.
Injector Seal (gold)	1 month or as needed	To ensure contamination-free chromatography and reduce (gold) analyte breakdown. Discoloration of the gold seal indicates contamination (Usually needed sooner than 1 month).
Injector Seal Washer	6 months or as needed	To ensure contamination-free chromatography.
Needle Valve	As needed	Auxiliary flow fluctuations are indicative of a faulty needle valve.
Pencil Trap	1 year or as needed	To ensure contamination-free chromatography.
Purge Valve	1 year or as needed	Poor linearity and random continuing standard linearity is indicative of a clogged purge valve; especially if the early and late eluting compounds of the standard are exhibiting exceedingly contrasting percent differences.
Rinse Vials	6 months or as needed	Rinse vial solvent should be filled and waste vials should be emptied daily. Do not leave lids off the waste vials. If contamination is suspected, rinse vials well with solvent and make a test injection. If the contamination persists, replace the vials.
Septum	Before every sequence	To ensure contamination-free chromatography. J&W Scientific "Ultimate Gold Septa", part number 701-0090, are designed for a high number of injections and may be changed after every 300 injections.
Septum Nut	1 year or as needed	To ensure contamination-free chromatography. Sonication the nut in methylene chloride, methanol, and hexane may preclude the need for replacement.
Split Line	6 months or as needed	To ensure contamination-free chromatography; also to prevent clogging of the purge valve with heavier organics back-flushed into the lines. Flushing the lines with methylene chloride, methanol, and hexane may preclude the need for replacement.
Syringe	1 month or as needed	To ensure contamination-free chromatography. Needed when the syringe plunger becomes difficult to move or when contamination traced to the syringe is noted.

TABLE 2
Miscellaneous Gas Chromatograph Preventative Maintenance Schedule

MAINTENANCE	TIME	COMMENT(S)
Auxiliary Flow Check	2 weeks or when a flow problem is suspected.	To ensure gas flow stability and to help foresee other problems.
Analytical/Guard Column Clipping	As needed	When analyte breakdown exceeds pesticide/PCB method acceptance criteria. When high baseline rise and/or peak tailing are believed to be caused by"dirt" at the front end of the column.
Column Flow Check	1 month or as needed	To ensure gas flow stability and to help foresee other problems.
Injection Port Rinse	As needed	To ensure contamination-free chromatography
Split Flow Check	As needed	To ensure gas flow stability, to prevent wasting gas, and to help foresee other problems.
Purge Flow Check	As needed	To ensure gas flow stability, to prevent wasting gas, and to help foresee other problems.
ECD	As needed	H_2 bake-out is recommended when the detector signal rises100% above the original signal, the baseline will not stabilize, or sample analyses have caused severe baseline rise. Cleaning the detector anode is also recommended when a great number of petroleum-containing samples have been analyzed.
NPD	As needed	Refurbishing/replacing of the bead is recommended when the detector loses stability (high drift) or when severe peak tailing precludes sound analyses. See refurbishing kit instructions for the procedure.
FID	2 weeks	Cleaning is required when the detector loses sensitivity. Cleaning on a routine basis is highly recommended to avoid chromatographic difficulties.
Gas-line Rinsing	As needed	Gas lines should be rinsed with methylene chloride, methanol, and hexane (in this order) after any period in which contaminated gas was suspected. Dry the lines with ultra pure nitrogen after solvent-flushing.

8.2 The following are recommended after every maintenance:

8.2.1 Leak-check the instrument around every area that was serviced. Thorough leak testing, including all possible points of leaking in the oven and surrounding anywhere near where the instrument was manipulated.

8.2.2 Complete the "GC Lab Instrument Maintenance Log". All work performed will be documented in the instrument maintenance log specific to that instrument. See Attachment 1 for an example log.

8.2.2.1 Document: Problems noted, maintenance performed, date of maintenance, date of return to analytical control, and initials of the one performing maintenance.

8.2.2.2 Hewlett-Packard service persons are also required to complete the instrument maintenance log (the person assigned that particular instrument is responsible for ensuring all appropriate entries).

8.2.3 Allow for equilibration time before beginning an analysis sequence. Before beginning an analytical sequence, inject a column conditioner and several solvents. Column conditioning promotes stability and is highly recommended.

8.2.4 Check detector signals for stability.

9.0 CALCULATIONS. N/A

10.0 QUALITY ASSURANCE/QUALITY CONTROL. N/A

11.0 WASTE DISPOSAL. See Standard Operating Procedure, "*Hazardous Waste Management Plan*", SWL-GA-114.

12.0 METHOD PERFORMANCE. N/A

13.0 REFERENCES

13.1 Standard Operating Procedure, "*Laboratory Safety Plan*", SWL-GA-111.

13.2 Standard Operating Procedure, "*Hazardous Waste Management Plan*", SWL-GA-114.

14.0 DEFINITIONS

ECD:..... Electron capture detector.

FID: ..... Flame ionization detector.

Instrument Maintenance Logbook: A permanently bound, sequentially numbered logbook used for detailing instrument problems, maintenance performed, and return to analytical control. One logbook must be kept for every instrument.

Leak testing: ..... A systematic search for carrier or auxiliary gas leaks in a pressurized gas chromatographic system. The Gow Mac, 21-250 gas detection system is used in the Gas Chromatography Laboratory. Follow all manufacturer's instructions.

NPD:..... nitrogen-phosphorus detector.

N/A:..... not applicable.

15.0 ATTACHMENTS

Attachment 1: ..... GC Lab Instrument Maintenance Log

ATTACHMENT 1
GC Lab Instrument Maintenance Log

GC Lab Instrument Maintenance Log

SWLO-OP-201 OP-201-MNTLOG-1 SWL Rev. #: _____

Maintenance performed and reason.	Date Maint. Performed	Analyst Initials	Date of Return to Analytical Control

Southwest Laboratory of Oklahoma, Inc.

SOUTHWEST LABORATORY OF OKLAHOMA, INC. *and*
AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.

Standard Operating Procedure

Analysis of Herbicides by GC, SW-846, Method 8151A

Document No.: SWL-OP-407

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ANALYSIS OF HERBICIDES BY GC, SW-846, METHOD 8151A

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1.0 SCOPE AND APPLICATION

1.1 This method is designed to determine the concentrations of various acid herbicides in extracts from solid and liquid matrices. The method is based on EPA SW-846 methods 8000B and 8151A for analysis, extractions and cleanups (Method 3580A is utilized for waste dilution). The method can be used to determine analyte concentrations of herbicides in the ppb range with the electron capture detector (ECD). Test codes encompassed by this method include but are not limited to GC550, GC560, GC570, GC571, GC580, and GC599.

1.2 Tables 1 and 2 list routinely analyzed herbicides using this method:

TABLE 1
Herbicide Target Compounds List (Full List)

COMPOUND NAME	CAS
2,4-D	94-75-7
2,4,-DB	94-82-6
2,4,5-TP (Silvex)	93-72-1
2,4,5-T	93-76-5
Dalapon	75-99-0
Dicamba	1918-00-9
Dichloroprop	120-36-5
Dinoseb	88-85-7
MCPA	94-74-6
MCPP	93-65-2
4-Nitrophenol	100-02-1
Pentachlorophenol	87-86-5

Table 2
Herbicide Pesticide Target Compounds List (Extended List)

COMPOUND NAME	CAS
2,4-D	94-75-7
2,4,-DB	94-82-6
2,4,5-TP (Silvex)	93-72-1
2,4,5-T	93-76-5
Dalapon	75-99-0
Dicamba	1918-00-9
Dichloroprop	120-36-5
Dinoseb	88-85-7
MCPA	94-74-6
MCPP	93-65-2
4-Nitrophenol	100-02-1
Pentachlorophenol	87-86-5
Acifluorfen	50594-66-6
Bentazon	25057-89-6
Chloramben	133-90-4
DCPA Diacid (a)	2136-79-0

(a) DCPA monoacid and diacid metabolites included in method scope; DCPA diacid metabolite used for validation studies. DCPA is a dimethyl ester.

1.3 This method utilizes wide bore capillary (0.53mm ID) or narrow bore capillary (0.32 or 0.25mm ID) columns for both the quantitation and confirmation analyses.

1.4 See the Redbook for quantitation limits (QLs) and method detection limits (MDLs).

1.5 This procedure contains no deviations from the method.

2.0 METHOD SUMMARY

2.1 Method 8151A provides extraction, derivatization and gas chromatographic conditions for the analysis of chlorinated acid herbicides in water, soil and waste samples. An option for the hydrolysis of esters is also described.

2.1.1 Water samples are extracted with diethyl ether and then esterified with diazomethane. The derivatives are determined by gas chromatography with an electron capture detector (GC/ECD). The results are reported as acid equivalents.

2.1.2 Soil and waste samples are extracted and esterified with diazomethane. The derivatives are determined by GC/ECD. The results are reported as acid equivalents.

2.1.3 If herbicide esters are to be determined using this method, hydrolysis conditions for the esters in water and soil extracts are described.

2.2 Two columns of different stationary phases are used for quantitation and confirmation of the analytes.

2.3 Columns are calibrated using the external calibration procedure with 5 concentrations of the analytes covering the working linear range of the ECD beginning from the level of quantitation or below. Calibration criteria specified in method 8000B are followed (unless otherwise specified).

2.4 Calibration verification standards are analyzed after every 10-20 samples and before the start of an analysis sequence so that they bracket the samples.

2.5 A method blank, laboratory control spike, and a matrix spike/matrix spike duplicate must be prepared for each batch of 20 samples or less per matrix.

2.6 All analytes tentatively identified within established retention windows on the quantitation column must be analyzed on a confirmation column. The lower analytical result is reported if the analyte is identified on both columns, and both columns are quantitative. Otherwise, the value from the acceptable quantitation column is reported.

3.0 HEALTH AND SAFETY

3.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method (see Standard Operating Procedure SWL-GA-111,

"*Laboratory Safety Plan*".) A reference file of Material Safety Data Sheets (MSDS) is available to all personnel involved in the chemical analysis.

3.2 Caution should be taken when working with all sample extracts and calibration standards. Samples are of unknown origin and should be considered hazardous. Many of the spiking solutions used, along with chlorinated solvents are considered carcinogens. Concentrated acids and bases, if not handled properly, can cause severe burns.

3.3 Gloves should be used for the manipulation of all sample extracts and calibration standards and preparation of all acid and base solutions. Wear personal protective equipment (PPE) and goggles or face shield when warranted. See the standard operating procedure SWL-GA-111, "*Laboratory Safety Plan*," for detailed safety procedures.

3.4 Diazomethane is a toxic carcinogen and can explode under certain conditions. The following precautions must be followed:

3.4.1 Use only a well-ventilated hood - do not breathe vapors.

3.4.2 Use a safety screen.

3.4.3 Use mechanical pipetting aides.

3.4.4 Do not heat above 90°C - EXPLOSION may result.

3.4.5 Avoid grinding surfaces, ground glass joints, sleeve bearings, glass stirrers - EXPLOSION may result.

3.4.6 Store away from alkali metals - EXPLOSION may result.

3.4.7 Solutions of diazomethane decompose rapidly in the presence of solid materials such as copper powder, calcium chloride, and boiling chips.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

4.1 Sample Extraction

4.1.1 Aqueous samples must be extracted within 7 days of collection. Solid samples must be extracted within 14 days of collection.

4.1.2 Extracts must be stored under refrigeration at 4°C (±2°C) and analyzed within 40 days of extraction.

4.2 Sample Storage

4.2.1 The samples must be protected from light and refrigerated at 4°C (±2°C) from the time of receipt until 60 days after delivery of a complete, reconciled sample data package to the client. After 60 days, the samples may be disposed of in a manner that complies with all applicable regulations.

4.2.2 The sample must be stored in an atmosphere demonstrated to be free of all potential contaminants.

4.2.3 Samples, sample extracts, and standards must be stored separately.

4.3 Sample Extract Storage

4.3.1 Sample extracts must be protected from light and stored at 4°C (±2°C).

4.3.2 Sample extracts must be stored in an atmosphere demonstrated to be free of all potential contaminants.

4.3.3 Samples, sample extracts, and standards must be stored separately.

4.4 All containers should be glass jars or bottles with Teflon lids. A minimum of one liter is required for aqueous samples (two liters is recommended). A minimum of 80g is required for soil samples, which includes enough additional sample for pH and percent moisture determination (125mL glass jar). One-hundred milliliters of TCLP extract is required for TCLP analysis.

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

5.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts or elevated baselines in gas chromatograms. All these materials must be analyzed routinely as reagent blanks to demonstrate the absence of interferences under the conditions of the analysis.

5.1.1 Glassware must be scrupulously cleaned. Clean each piece of glassware as soon as possible after use by rinsing it with the last solvent used. This should be followed by detergent washing with hot water and rinses with tap water, then with organic-free reagent water. Glassware should be solvent-rinsed with acetone and pesticide-quality hexane. After rinsing and drying, glassware should be sealed and stored in a clean environment to prevent any accumulation of dust or other contaminants. Store glassware inverted or capped with aluminum foil. Immediately prior to use, glassware should be rinsed with the next solvent to be used.

5.1.2 The use of high purity reagents and solvents helps to minimize interference problems. Purification of solvents by distillation in all-glass systems may be required.

5.2 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from sample to sample, depending upon the nature and diversity of the waste being sampled.

5.3 Organic acids, especially chlorinated acids, cause the most direct interference with the determination by methylation. Phenols, including chlorophenols, may also interfere with this procedure.

5.4 Alkaline hydrolysis and subsequent extraction of the basic solution removes many chlorinated hydrocarbons and phthalate esters that might otherwise interfere with the electron capture analysis. However, hydrolysis may result in the loss of dinoseb and

formation of aldol condensation products if any residual acetone remains from the extraction of soils.

5.5 The herbicides, being strong organic acids, react readily with alkaline substances and may be lost during analysis. Therefore, glassware must be acid-rinsed and then rinsed to constant pH with organic-free reagent water. Sodium sulfate must be acidified.

5.6 Sample extracts must be dry prior to methylation or else poor recoveries will be obtained.

6.0 EQUIPMENT/APPARATUS

Gas chromatograph — Analytical system complete with gas chromatograph suitable for on-column, direct, and split-splitless injection and all required accessories including syringes, analytical columns, gases, electron capture detector, and recorder/integrator or data system.

6.1 HP 5890 Gas Chromatograph with ECD detector(s) and a 7673 autosampler. The autosampler must be capable of 1 µL or 2 µL injections. The carrier gas should be ultra pure helium or hydrogen, and the makeup gas (detector gas) should be N_2.

6.2 Columns:

6.2.1 Column 1 — 30 m x 0.32 or 0.25 mm ID fused silica capillary column chemically bonded with 50 percent phenyl methylpolysiloxane (DB-17MS, or equivalent), 0.25 µm film thickness.

6.2.2 Column 2 — 30 m x 0.32 or 0.25 mm ID fused silica capillary column chemically bonded with approximately 14 percent phenyl methylpolysiloxane (DB-XLB, or equivalent), 0.25 µm film thickness.

6.2.3 Other dissimilar phase columns may be used as long as they meet acceptance criteria of the method.

7.0 REAGENTS

7.1 Solvents:

7.1.1 Methylene chloride, CH_2Cl_2. Pesticide quality or equivalent.

7.1.2 Acetone, CH_3COCH_3. Pesticide quality or equivalent.

7.1.3 Methanol, CH_3OH. Pesticide quality or equivalent.

7.1.4 Toluene, $C_6H_5CH_3$. Pesticide quality or equivalent.

7.1.5 Diethyl Ether, $C_2H_5OC_2H_5$. Pesticide quality or equivalent. Must be free of peroxides as indicated by test strips (EM Quant, or equivalent). Procedures for removal of peroxides are provided with the test strips. After cleanup, 20mL of ethyl alcohol preservative must be added to each liter of ether.

7.1.6 Isooctane, $(CH_3)_3CH_2CH(CH_3)_2$. Pesticide quality or equivalent.

7.1.7 Hexane, C_6H_{14}. Pesticide quality or equivalent.

7.1.8 Ethanol, absolute. C_2H_5OH.

7.1.9 Carbitol (diethylene glycol monoethyl ether), $C_2H_5OCH_2CH_2OCH_2CH_2O$-optional for producing alcohol free diazomethane.

7.2 All gases (i.e., carrier, auxiliary, etc.) should be at least high purity grade.

7.3 Standard Solutions:

7.3.1 Certified standard solutions (in the acid form) may be obtained from several sources, including Accustandard or Restek. It is recommended that these be used for traceability purposes.

7.3.2 Stock standard solutions are prepared by diluting a certified standard in Methanol.

7.3.3 Calibration Standards — A minimum of five calibration standards for each parameter of interest should be prepared by diluting and deriving the stock standard. One of the concentrations should be at or below the PQL. The remaining concentrations should bracket the expected range of concentrations found in real samples or should define the linear range of the GC/ECD. The components are diluted in hexane after derivitization (Section 8.1.5.3.7).

Table 3
Herbicide Calibration Standard (Full list) — Low Point Concentration

Low Point Concentration	ug/mL
2,4-D	0.25
2,4,-DB	0.45
2,4,5-TP (Silvex)	0.05
2,4,5-T	0.05
Dalapon	0.80
Dicamba	0.10
Dichloroprop	0.15
Dinoseb	0.10
MCPA	7.0
MCPP	7.0
4-Nitrophenol	0.25
Pentachlorophenol	0.05

Table 4
Herbicide Calibration Standard: (Extended list) — Low Point Concentration

Low Point Concentration	ug/mL
2,4-D	0.25
2,4,-DB	0.45
2,4,5-TP (Silvex)	0.05
2,4,5-T	0.05
Dalapon	0.80
Dicamba	0.10
Dichloroprop	0.15
Dinoseb	0.10
MCPA	7.0
MCPP	7.0
4-Nitrophenol	0.25
Pentachlorophenol	0.05
Acifluorfen	TBD
Bentazon	TBD
Chloramben	TBD
DCPA Diacid (a)	TBD
3,5-Dichlorobenzoic Acid	TBD
5-Hydroxydicamba	TBD
Picloram	TBD

Note: Level 2 concentration = 2 x Level 1; Level 3 concentration = 3 x Level 1; Level 4 concentration = 4 x Level 1; Level 5 concentration = 5 x Level 1

Keep stored at 4°C (±2°C) no longer than 6 months.

TBD = to be determined.

7.4.3 All solutions (standards, surrogates, and spikes) must be labeled, assigned a standards log number, and stored in a refrigerator protected from light as required by the standard operating procedure for "Standards Receipt, Traceability, and Preparation", SWL-OP-202. Solutions should be replaced after 6 months or sooner if comparison with check standards indicates a problem.

7.4.4 Surrogate standard — The surrogate solution of DCAA is prepared in methanol at a concentration of 15µg/mL. Label the standard and assign a standard log number. The standard can be used for up to 6 months when refrigerated in the dark. The surrogate is prepared so that when 0.5mL of the solution is spiked and brought to 10mLs final volume, the concentration will yield a mid-range response.

7.4.5 Matrix spike/Matrix Spike Duplicate — A spiking solution is prepared in methanol at concentrations shown in Table 5 so that a 1.0mL spike will give a mid-range response in the final extract.

Table 5
Herbicide Matrix Spiking Standard Concentration

COMPOUND	ug/mL
2,4-D	24.0
2,4-DB	13.5
2,4,5-TP (Silvex)	1.5
2,4,5-T	1.5
Dalapon	24.0
Dicamba	3.0
Dichloroprop	4.5
Dinoseb	3.0
MCPA	210
MCPP	210
4-Nitrophenol	7.5
Pentachlorophenol	1.5
Acifluorfen	TBD
Bentazon	TBD
Chloramben	TBD
DCPA Diacid (a)	TBD
3,5-Dichlorobenzoic Acid	TBD
5-Hydroxydicamba	TBD
Picloram	TBD

8.0 PROCEDURE

8.1 Derivation

8.1.1 Slowly blow extract down to ≈1mL.

8.1.2 Add 1mL of iso-octane.

8.1.3 Add 0.5mL of methanol.

8.1.4 Rinse vial with ether and swirl (should be one solution at this point.)

8.1.5 Add ≈2mL of diazomethane and swirl. Solution should turn yellow and stay yellow, if sample extract was clear. If extract does not remain yellow (this should happen for TCLP extracts only), add more diazomethane until it remains yellow.

8.1.6 Let stand for ≈15minutes, solution should still be yellow. Put an "X" on the label (to show extract has been derived.)

8.1.7 Slowly blow extract down to ≈2mL extract should turn clear (if it started that way.)

8.1.8 Bring up to 10mL total volume with hexane, extract is ready to analyze.

8.2 Instrument conditions: These conditions are only recommended starting conditions. Each instrument may vary and therefore require changes to be optimized.

8.2.1 Carrier gas hydrogen:
Head pressure: 5psi

8.2.2 Injector, Detector: 220°C, 320°C.
Splitless injection using Double Gooseneck liner
Injection Volume: 1 µL.

Detector gases: N_2 = 80-90 mL/min total.
Temperature Program: 50°C at 25°C/min to 150°C; 10/min to 200 for 2min: 11/min 280 for 1min

TCLP and RCRA extracts may be analyzed isothermally at 220°C.

8.3 Calibration

8.2.1 Refer to Method 8000B for proper calibration techniques. Percent relative standard deviation must be ≤ 20 for all analytes over the five-point calibration range to assume linearity through the origin (and use the mean calibration factor for quantitation). In those instances where the RSD for one or more analytes exceeds 20%, the initial calibration may still be acceptable for mean calibration factor use if the following conditions are met: 1) The mean of the RSD values for all analytes in the calibration is ≤ 20%. The mean RSD is calculated by summing the RSD value for each analyte and dividing by the total number of analytes. If no analyte has an RSD above 20%, then the mean RSD calculation need not be performed. 2) The mean RSD criterion applies to all analytes in the standard, regardless of whether or not they are of interest for a specific project. In other words, if the target analyte is part of the calibration standard, its RSD value is included in the evaluation. 3) The data user must be provided with either a summary of the initial calibration data or a specific list of those compound for which the RSD exceeded 20% and the results of the mean RSD calculation.

Linear fit calibration or non-linear (quadratic) calibration may be used if % RSD is > 20 and the mean RSD is also > 20% as long as the correlation coefficient or coefficient of determination (for quadratic) are 0.99 or greater.

Note: the low point calibration curve must be at or below the PQL.

8.2.2 Calculation of retention time windows follows method 8000B's default method because retention time windows established by this method are 0.00 to 0.01 (to the 1/100th of a minute) for all compounds when using the 72/72+ hour method. This is due to highly stable chromatographic systems. The use of such retention time windows for "real-world" samples may result in numerous false negatives, therefore, absolute windows of ± 0.03 minutes are used. 72-hour retention time studies (Form 9Es) are maintained for each instrument and column per method 8000B to prove the stability of the instruments.

8.2.3 The procedure of external calibration is used. Quantitation using the mean calibration factor is preferred, however, linear least squares and quadratic fit may be used. If a quadratic fit is to be used, six levels of standards must be analyzed for the initial calibration.

8.2.4 All confirmed values above the practical quantitation limit should be reported. Values below the PQL should be reported with the "J" flag if confirmed with a high degree of confidence. In some instances, analyst judgment is relied on heavily.

8.2.5 For quantitated hits above the PQL, a "P" flag is recommended to indicate the difference between quantitation and confirmation values exceeds 25%.

8.3 Gas chromatographic analysis:

8.3.1 Following acceptable initial calibration and calibration verification standards, samples may be analyzed. Analysis of a mid-concentration standard every 10 samples is recommended for dirty samples. Relatively clean samples may allow for an extended sequence (not to exceed a 12-hour period per Method 8000B) based on analyst judgment. Each sample must be bracketed with an acceptable initial calibration or calibration verification standards interspersed between the sample analyses. When a calibration verification standard fails to meet the ± 15% difference criteria, all samples that were injected after the last standard that last met the criteria must be re-injected. Several exceptions to this rule follows: 1) If the ending calibration verification bracket fails with percent differences > 15% above the initial calibration response and no "hits" are present in the samples, then the samples may be reported; 2) If the average of the responses for all analytes is within 15%, then the calibration has been verified. However, the average must include all analytes in the calibration, regardless of whether they are target analytes for a specific project, and the data user must be provided with the calibration verification data or a list of those analytes that exceeded the 15% limit. The effect of using the average of the response for all analytes for calibration verification would be similar to that for the initial calibration, namely that the quantitative results for those analytes where the difference is > 15% will include a greater uncertainty.

8.3.2 Examples of GC/ECD chromatograms generated by instruments with wide-bore capillary columns are presented in Attachments 2 and 3.

8.3.3 If the peak response is less than 2.5 times the baseline noise level, the validity of the quantitative result may be questionable. The analyst should consult with the source of the sample to determine whether further concentration of the sample is warranted by the context in which the result is to be used.

8.3.4 If the peak response exceeds the range of the highest standard, quantitatively dilute the extract and reanalyze.

8.3.5 Identify compounds in the sample by comparing the retention times of the peaks in the sample chromatogram with those of the peaks in standard chromatograms. Take into account the variance caused by matrix effect in samples.

8.3.6 Quantitation of the compound(s) of interest is premised on: 1) a linear response of the ECD to the ranges of concentrations of the compound(s) of interest encountered in the sample extract and the corresponding calibration; and 2) a direct linear proportionality between the magnitude of response of the ECD over the width(s) of the retention window(s). Proper quantitation requires the appropriate selection of a baseline from which the area under the characteristic peak(s) can be calculated.

8.3.7 If compound identification or quantitation are precluded due to interference (e.g., broad. rounded peaks or ill-defined baselines are present), cleanup of the extract or replacement of the capillary column or detector may be warranted.

8.4 See the standard operating procedure for the "Preventive Maintenance of Hewlett-Packard and Fisons Gas Chromatographs", SWL-OP-201. for specific GC maintenance procedures.

9.0 CALCULATIONS

9.1 Calibration Factor = $\frac{\text{Peak Area (or Height) of the Compound in the Standard}}{\text{Concentration of the Compound Injected (ng/mL)}}$

9.2 Mean CF = $\frac{\text{Sum of CF for each level}}{\text{number of levels}}$

9.3 SD = Square root $\left[\frac{\text{Sum of (CF of each individual level - Mean CF)}^2}{\text{(number of levels - 1)}}\right]$

9.4 %RSD = $\frac{\text{SD}}{\text{Mean CF}} \times 100$

9.5 See SW-846, Revision 2, December 1996, page 8000B - 20 through 23 for linear and higher order fit equations.

9.6 % Difference = $\frac{\text{Mean CF - CF from the calibration verification standard}}{\text{Mean CF}} \times 100$

9.7 % Drift (used for linear and higher order fits) = $\frac{\text{Calculated conc. - Nominal conc.}}{\text{Theoretical conc.}} \times 100$

9.8 Determine the concentration of individual compounds in the sample.

9.8.1 Sample Concentration Calculation:

9.8.1.1 Aqueous Samples

$$\text{Concentration (ug/L)} = \frac{[As]\,[V(t)][D]}{[\text{Mean CF}][V(s)][1000\ ng/\mu g]\,[1L/1000mL]}$$

where:
As = Area (or height) of the peak for the analyte in the sample.
V(t) = Total volume of concentrated extract (mL).
D = Dilution factor.
Mean CF = Mean calibration factor (mL/ng).
V(s) = Volume of aqueous sample extracted (mL).

NOTE: injection volume is not noted because it cancels out in the equation (the same injection volume is used for all samples, QC, and standards).

9.8.1.2 Non-aqueous Samples

$$\text{Concentration (ug/Kg)} = \frac{[As]\,[V(t)][D}{[\text{Mean CF}][W(s)][1000\ ng/\mu g]}$$

where:
As = Area (or height) of the peak for the analyte in the sample.
V(t) = Total volume of concentrated extract (mL).
D = Dilution factor.
Mean CF = Mean calibration factor.
W(s) = Dry Weight of sample extracted (Kg).

NOTE: injection volume is not noted because it cancels out in the equation (the same injection volume is used for all samples, QC, and standards).

9.9 Report results in ug/L or ug/Kg without correction for recovery data. All QC data obtained should be reported with the sample results.

9.10 Dry weight calculation (from data gathered during step 8.1.5.1.2):

$$\%\ \text{Moisture} = \frac{\text{grams of sample - grams of dried sample}}{\text{grams of sample}} \times 100$$

10.0 QUALITY ASSURANCE/QUALITY CONTROL

10.1 Every 20 samples or sample delivery group (SDG) is considered a batch. Two method blanks, laboratory control spikes, and a matrix spike (MS) and matrix spike duplicate (MSD) are extracted with each SDG of up to 20 samples. In most cases, a client will specify a sample for MS/MSD. If MS/MSD sample is not specified, the laboratory chooses one at their discretion. Also, if two or more clients send samples not totaling 20 in number, one set of method blanks, LCS and MS/MSD are applied to both clients' samples.

10.2 Any reagent blanks or matrix spike samples should be subjected to exactly the same analytical procedures as those used on actual samples.

10.3 Calculate surrogate standard recovery on all samples, blanks, and spikes. Determine if the recovery is within established limits (See SOP "Control Charts", SWL-GA-112 for more details). See "Blue Book" for SWLO's most current control limits. If recovery is not within limits, the following are required:

10.3.1 Check to be sure there are no errors in calculations or surrogate solutions. Also, check instrument performance.

10.3.2 Recalculate the data and/or reanalyze the extract if any of the above checks reveal a problem.

10.3.3 If the method blank recoveries are out of the advisory limits, reanalyze the sample to rule out instrument problems. Re-extract and reanalyze the samples if none of the above are a problem.

10.4 Include a mid-concentration calibration standard every 10 or more samples (not to exceed a 12-hour period) in the analysis sequence as a calibration check. The response factors for the mid-concentration calibration should be within 15 percent of the average values for the multi-concentration calibration. When this continuing calibration is out of this acceptance window, the laboratory should stop analyses, clean the injector, replace the septum and recalibrate the system. Samples that are not preceded by acceptable standards must be reinjected to avoid errors in quantitation. Several exceptions to this rule follows: 1) <u>If the ending calibration verification bracket fails with percent differences > 15% above the initial calibration response and no "hits" are present in the samples, then the samples may be reported;</u> 2) <u>If the average of the responses for all analytes is within 15%, then the calibration has been verified. However, the average must include all analytes in the calibration, regardless of whether they are target analytes for a specific project, and the data user must be provided with the calibration verification data or a list of those analytes that exceeded the 15% limit. The effect of using the average of the response for all analytes for calibration verification would be similar to that for the initial calibration, namely that the quantitative results for those analytes where the difference is > 15% will include a greater uncertainty.</u>

10.5 If any of the target analytes in the calibration verification fall outside of the retention time window established during initial calibration, the system is considered out of control. All samples analyzed after the last compliant calibration verification must be re-analyzed after instrument maintenance and recalibration is successfully performed.

10.6 Instrument blanks must not contain any confirmed target analyte greater ½ of the reporting limit for the associated samples. <u>Method blanks must not contain any confirmed target analyte greater than the reporting limit for the associated samples</u>. If contamination is noted, stop all analyses, determine the source of the contamination, remove the source, and confirm the source is gone by reanalyzing an instrument blank and/or method blank as appropriate. QC and associated samples will be re-extracted and reanalyzed if the blank contamination is present > PQL in the samples.

11.0 WASTE DISPOSAL/POLLUTION PREVENTION

See the Standard Operating Procedure for "*Hazardous Waste Management Plan*", SWL-GA-114.

12.0 METHOD PERFORMANCE

12.1 The MDL concentrations are updated annually and are obtained using organic-free

reagent water and sandy loam soil. The MDL results are maintained on file in the laboratory. Details for determining MDLs are given in the SOP "*Definition and Determination of Detection Limits*", SWL-GA-113. The MDL actually achievable in a given analysis will vary depending on detector response characteristics, irreducible noise from instrument electronics and matrix effects.

12.2 The accuracy and precision obtainable following this method will be determined by the sample matrix, sample preparation technique, optional cleanup techniques, and calibration procedures used.

12.3 The laboratory maintains control charts of recoveries and relative percent differences to judge accuracy and precision. See SOP "*Control Charts*", SWL-GA-112 for more details. See "Blue Book" for SWLO's most current control limits.

12.4 Quantitation Limits are based on the lowest calibration standard. SWLO's most current MDLs and QLs are found in the "Red Book".

13.0 REFERENCES

U.S.EPA SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods: "Method 3500B: Organic Extraction and Sample Preparation" Revision 2, December 1996.

U.S.EPA SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods: "Method 3580A. Waste Dilution" Revision 1, July 1992.

U.S.EPA SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods: "Method 8000B. Determinative Chromatographic Separations" Revision 2, December 1996.

U.S.EPA SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods: "Method 8151A. Chlorinated Herbicides by GC Using Methylation or Pentafluorobenzylation Derivatization" Revision 1. December 1996.

Standard Operating Procedure, "*Preventative Maintenance of Hewlett-Packard and Fisons Gas Chromatographs*", SWL-OP-201.

Standard Operating Procedure, "*Standards Receipt, Traceability, and Preparation*", SWL-OP-202.

Standard Operating Procedure, "*Laboratory Safety Plan*", SWL-GA-111.

Standard Operating Procedure, "*Control Charts*", SWL-GA-112.

Standard Operating Procedure, "*Definition and Determination of Detection Limits*", SWL-GA-113.

Standard Operating Procedure, "*Hazardous Waste Management Plan*", SWL-GA-114.

14.0 DEFINITIONS

PQL:..... The Practical Quantitation Limit.

N/A:..... Not applicable

Material Safety Data Sheet (MSDS): Information provided by commercial vendors for the chemical products prepared by their company. The information on the MSDS includes but is not limited to composition, physical properties, hazards identification, first aid measures, emergency clean-up and/or response, exposure, and storage.

SDG: ..... The Sample Delivery Group; a unit within a single case that is used to identify a group of samples for delivery. A SDG is a group of 20 or fewer field samples within a case, received over a period of up to 14 calendar days (7 calendar days for 14-day turns). Data from all samples in a SDG are due concurrently.

15.0 ATTACHMENTS

15.1 Attachment 1: Sample Extraction Log

15.2 Attachment 2: Herbicide Chromatogram

ATTACHMENT 1
Sample Extraction Log

[ ] RUSH [ ] REEXTRACT [ ] TCLP [ ]

SV B OTHER

XXXXX

SAMPLE EXTRACTION LOG

BATCH: 970617WE

EXTRACT DATE: 1997-06-17 11:57

SWLO
SOUTHWEST LABORATORY OF OKLAHOMA INC

LE ID ODE #)	CLIENT / CASE	DATE RECEIVED / CLIENT SAMPLE ID	MATRIX LEVEL	SAMPLE pH	% MOISTURE (%)	AMOUNT EXTRACTED (g or ml)	EXTRACTED VOLUME (ml)	GPC VOLUME (ml)	GPC ELUTE VOL (ml)	FLORISIL VOL CONC (ml)	FLORISIL FIN VOL CONC (ml)	VOL SV (ml)	FIN PROCESS VOLUME (ml)	SCREEN Y/N (Y/N)	MS SV (µl)	SURR SV (µl)	BASE pH CONFIRMATION	ACID pH CONFIRMATION	TOC < 1000 (Y/N)
617WE			W			1000	250					10.0	1.0	N		1000			N
617WE			W			1000	250					10.0	1.0	N		1000			N
617WE			W			1000	250					10.0	1.0	N	1000	1000			N
617WE			W			1000	250					10.0	1.0	N	1000	1000			N
34.01 17	AATS-E 25501	1997-06-12 15:15 X6236 (CASE #25501)	W			1000	250					10.0	1.0	N		1000	<2	<2	N
34.02 17	AATS-E 25501	1997-06-12 15:15 X6237 (CASE #25501)	W			1000	250					10.0	1.0	N		1000	<2	<2	N
35.01 17	XXXXXXXX XXXXXXXX	XXXXXXXXXXXXXXXX XXXXXXXXXXXXXXXXXXXX	W			1000	250					10.0	1.0	N		1000	<2	<2	N
35.02 17	XXXXXXXX XXXXXXXX	XXXXXXXXXXXXXXXX XXXXXXXXXXXXXXXXXXXX	W			1000	250					10.0	1.0	N		1000	<2	<2	N

INITIALS

GHING:	BL	EXTRACTION:	BL
ANUP:		KD:	BL
WDOWN:	BL	TV:	
ROG SPIKER:	BL	VERIFIER:	
RIX SPIKER:	BL	VERIFIER:	

ID NUMBERS

FLORISIL CARTRIDGE #:

GPC COLUMN #:

SURROGATES ID CONC #:

MATRIX SPIKE SOLN/CONC #(s):

METHYLENE CHLORIDE LOT #:

SODIUM SULFATE LOT #:

COMMENTS

ATTACHMENT 2
Herbicide Chromatogram

Quantitation Report

Signal #1 : F:\HPCHEM\HP\7\DATA\08_10_00\7_017308.D\ECD1A.CH Vial: 23
Signal #2 : F:\HPCHEM\HP\7\DATA\08_10_00\7_017308.D\ECD2B.CH
Acq On : 10 Aug 2000 23:19 Operator: JF
Sample : HERBL3 Inst : HP_07
Misc : 5-64-5 Dilution: 1.00
IntFile Signal #1: AUTOINT1.E IntFile Signal #2: autoint2.e
Quant Time: Aug 11 7:38 2000 Quant Results File: HERBH10.RES

Method : F:\HPCHEM\HP\7\METHODS\HERBH10.M
Title-Path : SW-846, METHOD 8151 - F:\HPCHEM\HP\7\DATA\08_04_00\
Last Update : Mon Aug 07 08:13:47 2000
Response via : Initial Calibration

Volume Inj. : 0.5 uL
Signal #1 Phase : DB-17ms Signal #2 Phase: DB-XLB
Signal #1 Info : .25 mm Signal #2 Info : .25 mm
Signal #1 Inst : HP_07A Signal #2 Inst : HP_07B

		Compound	RT#1	RT#2	Resp#1	Resp#2	ng/mL	ng/mL
		System Monitoring Compounds:						
3)	S	DCAA	12.70	11.93	3814659	1004646	678.4	683.5
		Target Compounds:						
1)	A	Dalapon	6.07	5.88f	11089397	1455911	2307.7	2265.3
2)	A	4-Nitrophenol	11.86	10.85	1543334	247523	684.1	685.3
[illegible]	A	Dicamba	13.12	12.11	4288400	1487336	270.4	269.0
[illegible]	A	MCPP	12.98	12.43	367098	100911	19175.4	19393.6
[illegible]	A	MCPA	13.66	12.80	909482	190598	18083.3	19772.1
7)	A	Dichloroprop	14.13	13.43	2202832	661837	408.3	410.6
8)	A	2,4-D	14.94	13.90	4226001	998104	739.5	690.2
9)	A	Pentachlorophenol	15.76	15.36	7705444	3252847	139.4	137.5
10)	A	2,4,5-TP(Silvex)	16.06	15.50	2952253	924952	145.6	139.0
11)	A	2,4,5-T	16.95	16.06	2830693	867620	140.8	139.2
12)	A	2,4-DB	17.63	16.90	4297444	721958	1256.5	1206.4
13)	A	Dinoseb	17.19	17.08	1747917	696175	287.9	264.8

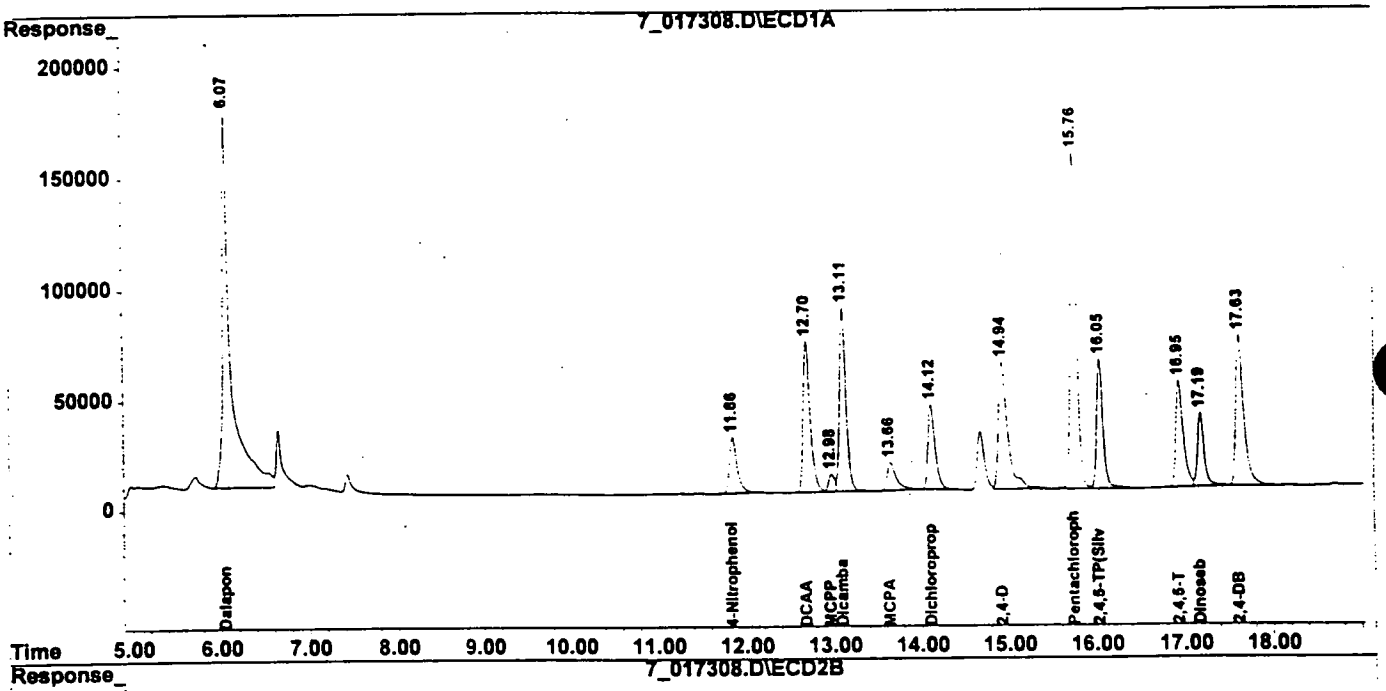
(f)=RT Delta > 1/2 Window (#)=Amounts differ by > 25% (m)=manual int.
(E)= > Highest cal. std. (d)=compound deleted (H) Dil. < med. std

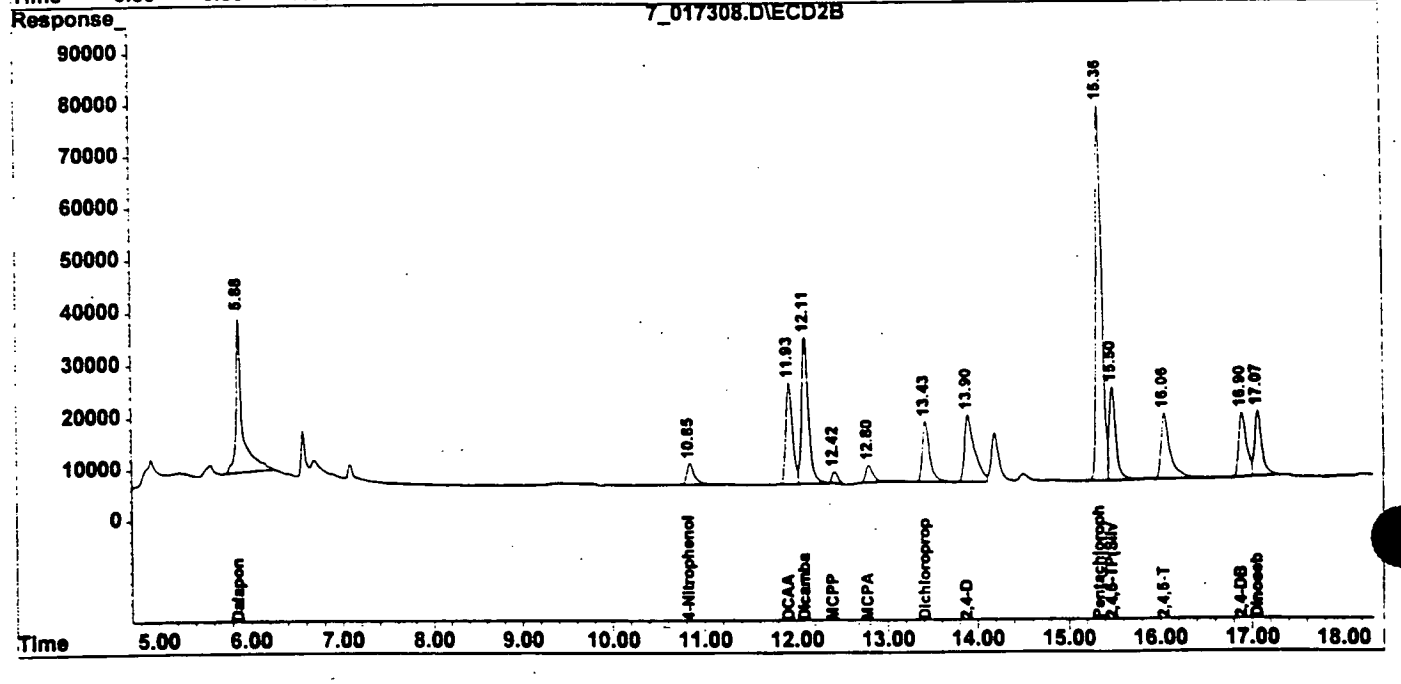
Chromatographic Report

Signal #1 : F:\HPCHEM\HP\7\DATA\08_10_00\7_017308.D\ECD1A.CH Vial: 23
Signal #2 : F:\HPCHEM\HP\7\DATA\08_10_00\7_017308.D\ECD2B.CH
Acq On : 10 Aug 2000 23:19 Operator: JF
Sample : HERBL3 Inst : HP_07
Misc : 5-64-5 Dilution: 1.00
IntFile Signal #1: AUTOINT1.E IntFile Signal #2: autoint2.e
Quant Time: Aug 11 7:38 2000 Quant Results File: HERBH10.RES

Method : F:\HPCHEM\HP\7\METHODS\HERBH10.M
Title-Path : SW-846, METHOD 8151 - F:\HPCHEM\HP\7\DATA\08_04_00\
Last Update : Mon Aug 07 08:13:47 2000
Response via : Multiple Level Calibration

Volume Inj. : 0.5 uL
Signal #1 Phase : DB-17ms Signal #2 Phase: DB-XLB
Signal #1 Info : .25 mm Signal #2 Info : .25 mm
Signal #1 Inst : HP_07A Signal #2 Inst : HP_07B





SOUTHWEST LABORATORY OF OKLAHOMA, INC. *and*
AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.

TM

Standard Operating Procedure

Analysis of Pesticides by GC, SW-846, Method 8081A

Document No.: SWL-OP-414

Rev No. / Date: Rev. 3.0 — 04/26/00

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APPROVALS	DATES
Procedure Prepared By	5/15/00 Date
Program Manager or Section Supervisor	5-15-00 Date
Laboratory Safety Officer	5/18/2000 Date
Laboratory QA/QC Officer	5/18/00 Date

(Effective Date is 5 calendar days after the last signature above - QA/QC Officer)

Document Status

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ANALYSIS OF PESTICIDES BY GC, SW-846, METHOD 8081A

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1.0 SCOPE AND APPLICATION

1.1 This method is designed to determine the concentrations of various organochlorine pesticides in extracts from solid and liquid matrices. The method is based on EPA SW-846 methods 8000B and 8081A. The method can be used to determine analyte concentrations of pesticides in the ppt range in water. Test codes encompassed by this method include but are not limited to GC801 and GC899.

1.2 A large number of compounds will give response in the electron capture detector (ECD) using this method. Tables 1 and 2 represent the compounds that may be analyzed by this method.

Table 1
Organochlorine Pesticide Target Compounds List

ANALYTE	CAS #
Aldrin	309-00-2
Alpha-BHC	319-84-6
Beta-BHC	319-85-7
Delta-BHC	319-86-8
Gamma-BHC	58-89-9
alpha-Chlordane	5103-71-9
gamma-Chlordane	5103-74-2
4,4'-DDD	72-54-8
4,4'-DDE	72-55-9
4,4'-DDT	50-29-3
Dieldrin	60-57-1
Endosulfan I	959-98-8
Endosulfan II	33212-65-9
Endosulfan Sulfate	1031-07-8
Endrin	72-20-8
Endrin Aldehyde	7421-93-4
Endrin Ketone	53494-70-5
Heptachlor	76-44-8
Heptachlor Epoxide	1024-57-3
4,4'-Methoxychlor	72-43-5
Toxaphene	8001-35-2

Table 2
Organochlorine Pesticide Target Compounds List (Extended List)

ANALYTE	CAS #
Aldrin	309-00-2
Alpha-BHC	319-84-6
Beta-BHC	319-85-7
Delta-BHC	319-86-8
Gamma-BHC	58-89-9
alpha-Chlordane	5103-71-9
gamma-Chlordane	5103-74-2
4,4'-DDD	72-54-8
4,4'-DDE	72-55-9
4,4'-DDT	50-29-3
Dieldrin	60-57-1
Endosulfan I	959-98-8
Endosulfan II	33212-65-9
Endosulfan Sulfate	1031-07-8
Endrin	72-20-8
Endrin Aldehyde	7421-93-4
Endrin Ketone	53494-70-5
Heptachlor	76-44-8
Heptachlor Epoxide	1024-57-3
4,4'-Methoxychlor	72-43-5
Toxaphene	8001-35-2
Chlorobenzilate	510-15-6
DBCP	96-12-8
Diallate	2303-16-4
Hexachlorobenzene	118-74-1
Hexachlorocyclopentadiene	77-47-4
Isodrin	465-73-6

NOTE 1: Some compounds may co-elute when analyzing for the extended list in one analysis.
NOTE 2: Technical chlordane may be analyzed in place of alpha- and gamma-chlordane upon client request.

1.3 This method utilizes wide bore capillary columns (0.53 mm ID) or narrow bore capillary (0.32 mm ID) columns for both the quantitation and confirmation analyses.

1.4 See the Redbook for most current quantitation limits (QLs).

1.5 There are no method deviations in this SOP.

2.0 METHOD SUMMARY

2.1 Continuous liquid-liquid or separatory funnel extraction procedures are employed for the extraction of aqueous samples. Sonication or Soxhlet extraction is required for soil/sediment samples. Gel Permeation (GPC) and florisil or alumina columns may be used for cleanup of extracts. Both neat and diluted organic liquids may be analyzed by direct injection (Method 3580A, Waste Dilution).

2.1.1 Continuous liquid-liquid extraction is not a recommended sample preparation method for Method 8081A analyses. Organochlorine pesticides many dechlorinate. Therefore, method 3510C is recommended for 8081A analyses. Method 3520C may be used in certain instances if pre-approved by management.

2.2 Samples are routinely analyzed on 2 columns of different stationary phases simultaneously to expedite the confirmation of target analytes. All calibration criteria apply to both columns independently. The lower analytical result (between the 2 columns) is routinely reported unless there is apparent evidence of negative matrix interference.

2.3 Columns are calibrated using the external calibration procedure with 5 concentrations of the analytes covering the working linear range of the ECD detectors beginning from the level of quantitation or below. Calibration criteria specified in method 8000B are followed (unless otherwise specified).

2.4 Calibration verification standards are analyzed each 12 hour shift (not to exceed 20 samples) and before the start of an analysis sequence so that they bracket the samples.

2.5 An evaluation standard containing both endrin and 4,4'-DDT is analyzed at the beginning of a calibration sequence and <u>at the beginning of each distinct analysis sequence</u> not to exceed a 12-hour shift. The evaluation standard monitors the breakdown of endrin and 4,4'-DDT.

2.6 A method blank, laboratory control spike, and a matrix spike/matrix spike duplicate must be prepared for all batches of 20 samples or less.

3.0 HEALTH AND SAFETY

3.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data Sheets (MSDS) is available to all personnel involved in chemical analysis.

3.2 The following parameters covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: 4,4'-DDT, 4.4'-DDD, the BHCs, and the PCBs. Primary standards of these toxic compounds <u>must</u> be prepared in a hood. A NIOSH/MESA approved toxic gas respirator <u>must</u> be worn when the analyst handles high concentrations of these toxic compounds.

3.3 Caution should be taken when working with all sample extracts and calibration standards. Samples are of unknown origin and should be considered hazardous. Many of the spiking solutions used, along with chlorinated solvents, are considered carcinogens. Concentrated acids and bases, if not handled properly, can cause severe burns.

3.4 Gloves should be used for the manipulation of all sample extracts and calibration standards and during the preparation of all acid and base solutions. Wear personal protective equipment (PPE) and goggles or face shield when warranted. See the standard operating procedure for "Laboratory Safety Plan", SWL-GA-111 for detailed safety procedures.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

4.1 Sample Extract Storage

4.1.1 Sample extracts must be protected from light and stored at 4°C (±2°C) until 365 days after delivery to the client of a complete data package.

4.1.2 Sample extracts must be stored in an atmosphere demonstrated to be free of all potential contaminants.

4.1.3 Samples, sample extracts, and standards must be stored separately.

4.2 All containers should be glass jars or bottles with Teflon lids. A minimum of one liter is required for aqueous samples. A minimum of 80 g is required for soil samples (125-mL glass jar).

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

5.1 Sources of interference in this method can be grouped into three broad categories: contaminated solvents, reagents or sample processing hardware; contaminated GC carrier gas, parts, column surfaces or detector surfaces; and the presence of co-eluting compounds in the sample matrix to which the ECD will respond. Knowledge of good laboratory practices is assumed, including steps to be followed in routine testing and cleanup of solvents, reagents and sample processing hardware, and instrument maintenance. The discussion that follows focuses on sources of interference associated with the sample matrix and compound classes that represent common sources of interference, particularly phthalate esters, organosulfur compounds, lipids, and waxes. Interferences co-extracted from the samples will vary considerably from sample to sample. While general cleanup techniques are referenced or provided as part of this method, unique samples may require additional cleanup approaches to achieve desired degrees of discrimination and quantitation.

5.2 Interferences by phthalate esters introduced during sample preparation can pose a major problem in pesticide determinations. These materials may be removed prior to analysis using Gel Permeation Chromatography (GPC) - pesticide option (Method 3640) or as Fraction III of the silica gel cleanup procedure (Method 3630). Common flexible plastics contain varying amounts of phthalate esters that are easily extracted or leached from such materials during laboratory operations. Cross-contamination of clean glassware routinely occurs when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled. Interferences from phthalate esters can best be minimized by avoiding contact with all plastic materials and checking all solvents and reagents for

phthalate contamination. Exhaustive cleanup of solvents, reagents and glassware may be required to eliminate background phthalate ester contamination.

5.3 The presence of elemental sulfur will result in large peaks that interfere with the detection of later eluting organochlorine pesticides. Method 3660 is suggested for removal of sulfur.

5.4 Waxes, lipids, and other high molecular weight co-extractables can be removed by GPC.

6.0 EQUIPMENT/APPARATUS

6.1 Gas chromatograph — Analytical system complete with gas chromatograph suitable for on-column, direct, and split-splitless injection and all required accessories including syringes, analytical columns, gases, electron capture detector, and recorder/integrator or data system.

6.1.1 HP 5890 Gas Chromatograph with ECD detector(s) and a 7673 autosampler. The autosampler must be capable of 1uL or 2uL injections. The carrier gas should be ultra pure helium or hydrogen, and the makeup gas (detector gas) should be ultra pure nitrogen or 5% methane / 95% argon.

The following columns pairs are recommended:

Column pair 1 — DB-17MS, DB-XLB fused silica capillary columns, 0.25mm ID x 30m, 0.25um film thickness.

Column pair 2 — RTX CLPesticides, 30m x 0.32mm ID, 0.5um film thickness, or 15m x 0.25mm ID, 0.25 film thickness. RTX CLPesticides II, 30m x 0.32mm ID, 0.25um film thickness, or 15m x 0.25mm ID, 0.25um film thickness.

Column pair 3 — DB17/1701P, 30m x 0.32mm ID, 0.25um film thickness.

6.1.2 GC injection ports are of critical concern, especially in the analysis of DDT and Endrin. Injectors that are contaminated, chemically active, or too hot can cause the degradation ("breakdown") of the analytes. Endrin and DDT break down to endrin aldehyde, endrin ketone, and DDD and DDE, respectively. When such breakdown is observed, replace inlet line, and if necessary clip one to three feet off guard column and remount it. A performance evaluation standard is analyzed at the beginning of each 12-hour period. See Table 6 for preparation.

6.1.3 HP Chemstation/HP EnviroQuant data system for acquisition/processing of chromatography data. Each system is capable of simultaneously controlling 4 dual detector gas chromatographs or equivalent.

7.0 REAGENTS

7.1 Reagent or pesticide grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are

available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 Standards preparation:

Prepare individual stock standard solutions in n-hexane or iso-octane at the following concentrations every six months or sooner if the solution has degraded or concentrated (Assign log numbers to each of the solutions and log them into the Standards Log Book; see Standard Operating Procedure for "Standards Receipt, Traceability, and Preparation", SWL-OP-202).

7.2.1 Stock Standard Solutions - Individual solutions are obtained from AccuStandard or equivalent supplier at concentrations of 1000 µg/mL in hexane.

7.2.1.1 Concentrated and certified standard mixtures can be obtained from several sources. For example, Restek and Supelco supply the pesticide mixtures. It is recommended that these be used for traceability purposes. Stock standard solutions can then be diluted directly from the concentrates.

7.2.1.2 The pesticides can be made in two solutions Individual A (INDA) and Individual B (INDB), or in one solution which has all of the compounds that are in the Individual A and Individual B mixes (INDAB).

7.2.1.3 Stock standard solutions must be kept refrigerated at 4°C (±2°C) for no longer than 6 months.

7.2.2 Calibration Standards — A minimum of five calibration standards for each parameter of interest (except multi-components which use a single point calibration at the PQL) should be prepared through dilution of the highest level standard with hexane or iso-octane. One of the concentrations should be at or below the PQL. The remaining concentrations should bracket the expected range of concentrations found in real samples or should define the working range of the GC/ECD. The components are diluted in hexane or iso-octane. Concentrations are in ng/mL.

7.2.2.1 Prepare solutions of the calibration standards for INDA and INDB or INDAB at 5 concentration levels beginning at the quantitation limit and spanning the linear working range of the detector. See Tables 3 and 4 for the solution concentrations. See Table 5 for the extended list of compounds. Multi-component standard concentrations may be seen in Tables 6 and 7. Table 7 lists the performance evaluation standard concentrations.

7.2.2.2 Calibration standards must be remade at least every six months, or verified against new standards before use.

Table 3
Pesticide Calibration Standards

Individual Standard Mixture A	Low Point Concentration ng/mL (Level 1)
alpha-BHC	3.75
Heptachlor	3.75
gamma-BHC	3.75
Endosulfan I	3.75
Dieldrin	7.5
Endrin	7.5
4,4'-DDD	7.5
4,4'-DDT	7.5
Methoxychlor	37.5
Tetrachloro-m-xylene	3.75
Decachlorobiphenyl	7.5

Table 4

Individual Standard Mixture B	Low Point Concentration ng/mL (Level 1)
beta-BHC	3.75
delta-BHC	3.75
Aldrin	3.75
Heptachlor epoxide	3.75
alpha-Chlordane	3.75
gamma-Chlordane	3.75
4,4'-DDE	7.5
Endosulfan sulfate	7.5
Endrin aldehyde	7.5
Endrin ketone	7.5
Endosulfan II	7.5
Tetrachloro-m-xylene	3.75
Decachlorobiphenyl	7.5

Note: Level 2 concentration = 2 x Level 1 Level 3 concentration = 4 x Level 1
Level 4 concentration = 8 x Level 1 Level 5 concentration = 16 x Level 1

Prepare all solutions in hexane or isooctane. Keep stored at 4°C (±2°C) no longer than 6 months.

Table 5

Individual Standard Mixture AB	Low Point Concentration ng/mL (Level 1)
TCX	3.75
alpha-BHC	3.75
gamma-BHC (Lindane)	3.75
beta-BHC	3.75
delta-BHC	3.75
Heptachlor	3.75
Aldrin	3.75
Heptachlor Epoxide	3.75
gamma-Chlordane	3.75
alpha-Chlordane	3.75
Endosulfan I	3.75
4,4'-DDE	7.5
Dieldrin	7.5
Endrin	7.5
Endosulfan II	7.5
4,4'-DDD	7.5
4,4'-DDT	7.5
Endrin Aldehyde	7.5
Methoxychlor	37.5
Endosulfan Sulfate	7.5
Endrin Ketone	7.5
DCB	3.75

Table 6

Extended List Mixture	Low Point Concentration ng/mL (Level 1)
Chlorobenzilate	37.5
DBCP	TBD
Diallate	72.5
Hexachlorobenzene	TBD
Hexachlorocyclopentadiene	TBD
Isodrin	3.75

TBD = to be determined

Note: Level 2 concentration = 2 x Level 1 Level 3 concentration = 4 x Level 1
Level 4 concentration = 8 x Level 1 Level 5 concentration = 16 x Level 1

Prepare all solutions in hexane or isooctane. Keep stored at 4°C (±2°C) no longer than 6 months.

Table 7

Multi-response calibration standards	Concentration (ng/mL)
Toxaphene	250
Technical Chlordane	10.0

Prepare all solutions in hexane or isooctane. Keep stored at 4°C (±2°C) no longer than 6 months.

Table 8

Performance Evaluation Mixture	(ng/mL)
gamma-BHC	10.0
alpha-BHC	10.0
beta-BHC	10.0
4,4'-DDT	100.0
Endrin	50.0
Methoxychlor	250.0
Tetrachloro-m-xylene	20.0
Decachlorobiphenyl	20.0

Performance Evaluation Mixture (PEM) - prepare the PEM in hexane or isooctane at the concentration levels listed below. The PEM must be prepared every six months or more often if the solution has degraded or concentrated.

7.2.3 Spiking Solutions:

7.2.3.1 Surrogate Solution

The surrogates, Tetrachloro-m-xylene and Decachlorobiphenyl, are added to all standards, samples, matrix spikes, and blanks. Prepare a surrogate spiking solution of 0.2 µg/mL of each of the two compounds in acetone or methanol. The solution should be checked frequently for stability. The solution must be replaced after six months, or sooner, if comparison with quality control check samples indicate a problem.

Caution: Analysts must allow all spiking solutions to equilibrate to room temperature before use.

7.2.3.2 Pesticide Matrix Spiking Solution

7.2.3.2.1 Prepare a spiking solution in methanol (acetone produces interfering aldol-condensation products) that contains the pesticides in the concentrations specified in Table 8.

Table 9
Matrix Spiking Solution (µg/mL)

ANALYTE	Concentration (µg/mL)
Aldrin	0.25
Alpha-BHC	0.25
Beta-BHC	0.25
Delta-BHC	0.25
Gamma-BHC	0.25
alpha-Chlordane	0.25
gamma-Chlordane	0.25
4,4'-DDD	0.25
4,4'-DDE	0.25
4,4'-DDT	0.25
Dieldrin	0.25
Endosulfan I	0.25
Endosulfan II	0.25
Endosulfan Sulfate	0.25
Endrin	0.25
Endrin Aldehyde	0.25
Endrin Ketone	0.25
Heptachlor	0.25
Heptachlor Epoxide	0.25
4,4'-Methoxychlor	1.0
Extended List Compounds:	
Chlorobenzilate	1.0
DBCP	TBD
Diallate	2.0
Hexachlorobenzene	TBD
Hexachlorocyclopentadiene	TBD
Isodrin	0.25

7.2.3.2.2 The solution must be prepared every six months or sooner if the solution has degraded or concentrated.

7.2.3.3 Florisil Cartridge Check Solution

7.2.3.3.1 Florisil cleanup is not performed as a routine, but clients may occasionally request its use. Before use, pesticide recoveries are verified through use of the following check solution. Table 9 displays the check solution components and concentrations.

Table 10
Florisil Check Solution (µg/mL)

Analyte	Concentration (µg/mL)
2,4,5-Trichlorophenol	0.10
Alpha-BHC	0.02
Gamma-BHC	0.02
4,4'-DDD	0.04
4,4'-DDT	0.04
Dieldrin	0.04
Endosulfan I	0.04
Endrin	0.04
Heptachlor	0.02
4,4'-Methoxychlor	0.2
TCX	0.02
DCB	0.04

7.2.3.3.2 The check solution should exhibit 80 - 120% recovery of the pesticides and less than 5% recovery of 2,4,5-TCP.

7.2.3.3.3 Store all standard solutions in amber glass bottles or vials with a Teflon-lined screw cap at 4°C (±2°C) and protect from light.

8.0 PROCEDURE

8.1 Analysis by Gas Chromatography

8.1.1 Gas Chromatograph Operating Conditions

Column Conditions:

DB-17MS/XLB

Carrier Gas:	Hydrogen at 12psi
Injector Temp:	220
Detector Temp:	320
Initial Temp:	110, hold for 0.5min.
Temperature Prog:	110 to 150 at 25deg/min
	150 to 250 at 9deg/min
	250 to 320 at 25deg/min
Final Temp:	320 hold for 3.0min

DB-17/1701

Carrier Gas:	Hydrogen at 7psi
Injector Temp:	220
Detector Temp:	320
Initial Temp:	110, hold for 0.5min
Temperature Prog:	110 to 140 at 20deg/min
	140 to 280 at 12deg/min
Final Temp:	280 hold for 6.33min

RTX-CLPesticides/CLPesticides II 30m x .32mm

Carrier Gas:	Hydrogen at 7psi
Injector Temp:	220
Detector Temp:	320
Initial Temp:	140, hold for 0.5min
Temperature Prog:	140 to 250 at 9deg/min
	250 to 300 at 15eg/min
Final Temp:	300 hold for 4.44min

RTX-CLPesticides/CLPesticides II 15m x .25mm

Carrier Gas:	Helium at 13psi
Injector Temp:	220
Detector Temp:	320
Initial Temp:	110, hold for 0.5min
Temperature Prog:	110 to 260 at 7.5deg/min
	260 to 290 at 1deg/min
Final Temp:	290 hold for 2.0min

DB-17/1701

Carrier Gas:	Helium at 11psi
Injector Temp:	220
Detector Temp:	320
Initial Temp:	110, hold for 0.5min
Temperature Prog:	110 to 140 at 20deg/min
	140 to 280 at 12deg/min
Final Temp:	280 hold for 6.5min

Optimize GC conditions for analyte separation and sensitivity. Once optimized, the same GC conditions must be used for the analysis of all standards, samples, blanks, and MS/MSDs.

8.1.2 Calibration

8.1.2.1 Refer to Method 8000B for proper calibration techniques. Percent relative standard deviation must be ≤ 20 for all analytes over the five-point calibration range to assume linearity through the origin (and use the mean calibration factor for quantitation). In those instances where the RSD for one or more analytes exceeds 20%, the initial calibration may still be acceptable for mean calibration factor use if the following conditions are met: 1) The mean of the RSD values for all analytes in the calibration is ≤ 20%. The mean RSD is calculated by summing the RSD value for each analyte and dividing by the total number of analytes. If no analyte has an RSD above 20%, then the mean RSD calculation need not be performed. 2) The mean RSD criterion applies to all analytes in the standard, regardless of whether or not they are of interest for a specific project. In other words, if the target analyte is part of the calibration standard, its RSD value is included in the evaluation. 3) The data user must be provided with either a summary of the initial calibration data or a specific list of those compound for which the RSD exceeded 20% and the results of the mean RSD calculation.

Linear fit calibration or non-linear (quadratic) calibration may be used if % RSD is > 20 and the mean RSD is also > 20% as long as the correlation coefficient or coefficient of determination (for quadratic) are 0.99 or greater. Note: the low point calibration curve must be at or below the PQL.

8.1.2.2 The procedure of external calibration is used. Quantitation using the mean calibration factor is preferred, however, linear least squares and quadratic fit may be used. If a quadratic fit is to be used, six levels of standards must be analyzed for the initial calibration.

8.1.2.3 SWLO's policy is to analyze a five point initial calibration for all pesticide analytes and a single-point calibration for all multi-component analytes (i.e., toxaphene, etc.). If a multi-component analyte is identified in a sample, then the single point calibration is used to obtain a quantitative value for the sample.

8.1.2.4 Because of the low concentration of pesticide standards injected on a GC/ECD, column adsorption may be a problem when the GC has not been used for a day. Therefore, the GC column should be primed or deactivated by injecting a pesticide standard mixture approximately 4 times more concentrated than the mid-concentration standard. Inject this standard mixture plus 10 to 15 solvent rinses prior to beginning initial or daily calibration, especially if the instrument has set for more than several hours.

Caution: Several analytes, including Aldrin, may be observed in the injection just following this system priming. Always run an acceptable blank prior to running any standards or samples.

8.1.2.5 A "P" flag is used to indicate when the difference between quantitation and confirmation values exceeds 40%.

8.1.3 Gas chromatographic analysis:

8.1.3.1 Following acceptable initial calibration and calibration verification standards, samples may be analyzed. Analysis of a mid-concentration standard every 10 samples is recommended for dirty samples. Relatively clean samples may allow for an extended sequence (not to exceed a 12-hour period per Method 8000B) based on analyst judgment. Each sample must be bracketed with an acceptable initial calibration or calibration verification standards interspersed between the sample analyses. When a calibration verification standard fails to meet the ± 15% difference criteria, all samples that were injected after the last standard that last met the criteria must be re-injected. Several exceptions to this rule follow: 1) <u>If the ending calibration verification bracket fails with percent differences > 15% above the initial calibration response and no "hits" are present in the samples, then the samples may be reported; 2) If the average of the responses for all analytes is within 15%, then the calibration has been verified. However, the average must include all analytes in the calibration, regardless of whether they are target analytes for a specific project, and the data user must be provided with the calibration verification data or a list of those analytes that exceeded the 15% limit. The effect of using the average of the response for all analytes for calibration verification would be similar to that for the initial calibration, namely that the quantitative results for those analytes where the difference is > 15% will include a greater uncertainty.</u>

8.1.3.2 Examples of GC/ECD chromatograms generated by instruments with capillary columns are presented in Attachments 2-5.

8.1.3.3 If the peak response exceeds the range of the highest standard, quantitatively dilute the extract and reanalyze.

8.1.3.4 Identification of mixtures (i.e. toxaphene, etc.) is based on the characteristic "fingerprint" retention time and pattern of the indicator peak(s); quantitation is based on the area under the characteristic peaks as compared to the area under the corresponding calibration peak(s) of the same retention time and pattern generated using external calibration procedures.

8.1.3.5 Identify compounds in the sample by comparing the retention times of the peaks in the sample chromatogram with those of the peaks in

standard chromatograms. The retention time windows used to make identifications are the retention time windows listed in Table 9. Take into account the variance caused by matrix effect in samples.

Table 11
Retention Time Windows

ANALYTE	RT Window (± minutes)
Aldrin	0.03
Alpha-BHC	0.03
Beta-BHC	0.03
Delta-BHC	0.03
Gamma-BHC	0.03
alpha-Chlordane	0.03
gamma-Chlordane	0.03
4,4'-DDD	0.03
4,4'-DDE	0.03
4,4'-DDT	0.03
Dieldrin	0.03
Endosulfan I	0.03
Endosulfan II	0.03
Endosulfan Sulfate	0.03
Endrin	0.03
Endrin Aldehyde	0.03
Endrin Ketone	0.03
Heptachlor	0.03
Heptachlor Epoxide	0.03
4,4'-Methoxychlor	0.03
Toxaphene	0.03
Chlorobenzilate	0.03
DBCP	0.03
Diallate	0.03
Hexachlorobenzene	0.03
Hexachlorocyclopentadiene	0.03
Isodrin	0.03
TCX	0.03
DCB	0.05

8.1.3.6 Quantitation of the compound(s) of interest is premised on: 1) a linear response of the ECD to the ranges of concentrations of the compound(s) of interest encountered in the sample extract and the corresponding calibration extract; and 2) a direct linear proportionality between the magnitude of response of the ECD over the width(s) of the retention window(s) (the area under the characteristic or "fingerprint" peak[s]) in the sample and calibration extracts. Proper quantitation requires the appropriate selection of a baseline from which the area under the characteristic peak(s) can be calculated.

8.1.3.7 If compound identification or quantitation are precluded due to interference (e.g., broad, rounded peaks or ill-defined baselines are present), cleanup of the extract or replacement of the capillary column or detector may be warranted.

8.1.4 Quantitation of Multiple Component Analytes:

8.1.4.1 Residues that are mixtures of two or more components present problems in measurement. When they are found together, e.g.,

Toxaphene and DDT, the problem of quantitation becomes even more difficult. Suggestions are offered in the following sections for handing Toxaphene.

8.1.4.2 Toxaphene:

8.1.4.2.1 Quantitative calculation of Toxaphene is difficult, but reasonable accuracy can be obtained. To calculate Toxaphene on GC/ECD, quantitate using the average of four major peaks or the total area of the Toxaphene pattern. The integration should follow the baseline trend and not follow the upward trend caused by Toxaphene.

8.1.4.2.2 When single-component analytes and Toxaphene are present in the same sample, quantitation is difficult. In-house studies have shown that single-component pesticides tend to "ride" on top of the Toxaphene "hump". Reasonable accuracy can be obtained by following the rise caused by Toxaphene when integrating the single-component compounds.

8.1.4.3 Technical Chlordane:

8.1.4.3.1 To calculate technical chlordane on a GC/ECD, quantitate using the average of three to five major peaks. The integration should follow the general baseline trend.

8.1.4.3.2 Technical chlordane component ratios may vary from manufacturer to manufacturer and may degrade significantly in the environment. Highly degraded chlordane looks similar to degraded Aroclor 1254. Therefore, special care must be taken in its identification.

8.1.5 See the standard operating procedure for the "Preventive Maintenance of Hewlett-Packard and Fisons Gas Chromatographs", SWL-OP-201, for specific GC maintenance procedures.

8.2 Sulfur Cleanup:

Sulfur Cleanup (Mercury Technique): Add one to three drops of mercury to each hexane extract in a clean vial. Tighten the top on the vial and agitate the sample for up to 30 minutes. Filter or centrifuge the extract. Pipette the extract to another vial and leave all solid precipitate and liquid mercury. If the mercury appears shiny, proceed with analysis. If the mercury turns black, repeat sulfur removal as necessary. The extract transferred to the vial still represents the same final volume.

9.0 CALCULATIONS

9.1 Calibration Factor (CF) = $\frac{\text{Peak Area or Height of the Compound in the Standard}}{\text{conc. in ng/mL of compound}}$

9.2 Mean CF = $\frac{\text{Sum of CF for each level}}{\text{number of levels}}$

9.3 SD = Square root $\left[\frac{\text{Sum of (CF of each individual level - Mean CF)}^2}{\text{(number of levels - 1)}}\right]$

9.4 RSD = $\frac{\text{SD}}{\text{Mean CF}} \times 100$

9.5 See SW-846, Revision 2, January 1995, page 8000B - 20 through 22 for linear and higher order fit equations.

9.6 % Difference = $\frac{\text{Mean CF - CF from the calibration verification standard}}{\text{Mean CF}} \times 100$

9.7 % Drift (used for linear and higher order fits) = $\frac{\text{Calculated conc. - Nominal conc.}}{\text{Theoretical conc.}} \times 100$

9.8 Calculate percent breakdown as follows:

$$\text{\% Breakdown DDT} = \frac{\text{Area of (DDD + DDE)}}{\text{Area of DDT + Area DDD + Area DDE}} \times 100$$

$$\text{\% Breakdown Endrin} = \frac{\text{Area of (Endrin aldehyde + Endrin ketone)}}{\text{Area of Endrin aldehyde + Area of Endrin ketone + Area of Endrin}} \times 100$$

9.9 Report results in µg/L for water samples and µg/Kg for solid samples without correction for recovery data. All QC data obtained should be reported with the sample results.

9.10 Dry weight calculation (from data gathered during step 8.1.5.1.6):

$$\% \text{ Moisture} = \frac{\text{grams of sample - grams of dried sample}}{\text{grams of sample}} \times 100$$

10.0 QUALITY ASSURANCE/QUALITY CONTROL

10.1 Refer to Chapter One of SW-846 for specific quality control (QC) procedures. Quality control to validate sample extraction is covered in Method 3500 and in the extraction method utilized. If extract cleanup was performed, follow the QC in Method 3600 and in the specific cleanup method.

10.2 4,4'-DDT and endrin are easily degraded in the injection port, if the injection port or front of the column is contaminated with buildup of high boiling residue from sample injection. Inject a mid-concentration standard containing 4,4'-DDT and endrin. Look for the degradation products 4,4'-DDE, 4,4'-DDD, endrin ketone, and endrin aldehyde. If degradation of either 4,4'-DDT or endrin exceeds **15%**, take corrective action according to the Standard Operating Procedure, "Preventative Maintenance of Hewlett-Packard and Fisons Gas Chromatographs", SWL-OP-201, before proceeding with calibration. See section 9.0, Calculations, for the procedure for calculating % breakdown.

10.3 Calculate surrogate standard recovery on all samples, blanks, and spikes. Determine if the recovery is within limits established. If recovery is not within limits, the following are required:

10.3.1 Check to be sure there are no errors in calculations and surrogate solutions. Also, check instrument performance.

10.3.2 Recalculate the data and/or reanalyze the extract if any of the above checks reveal a problem.

10.3.3 If both method blank surrogate recoveries are outside of control limits, reanalyze the sample to rule out instrument problems. Re-extract and reanalyze the samples if none of the above are a problem.

10.4 If greater than 50% of the spike recoveries are outside of laboratory control limits in both the LCS and LCSD or any spike recovery is below 10%, the LCS/LCSD and associated samples must be re-extracted and reanalyzed. If out-of-control analytes are out high and those analytes are not present in the samples, then no corrective action is required.

Control limits will be those annually updated from in-house generated data. See "Blue Book" for SWLO's current control limits.

10.5 If spike recoveries in the MS/MSD are outside of control limits but the combined LCS/LCSD are within control limits, the poor MS/MSD recoveries may be attributed to matrix effect. If both LCS/LCSD and MS/MSD have greater than 50% of the spiked

compounds outside of control limits, the samples and associated QC must be re-extracted and reanalyzed. Control limits will be those annually updated from in-house generated data. See "Blue Book" for SWLO's current control limits.

10.6 Include a mid-concentration calibration standard every 10 or more samples (not to exceed a 12-hour period) in the analysis sequence as a calibration check. The response factors for the mid-concentration calibration should be within 15 percent of the average values for the multi-concentration calibration. When this continuing calibration is out of this acceptance window, the laboratory should stop analyses, clean the injector, replace the septum and recalibrate the system. Samples that are not preceded by acceptable standards must be re-injected to avoid errors in quantitation. Several exceptions to this rule follows: 1) If the ending calibration verification bracket fails with percent differences > 15% above the initial calibration response and no "hits" are present in the samples, then the samples may be reported; 2) If the average of the responses for all analytes is within 15%, then the calibration has been verified. However, the average must include all analytes in the calibration, regardless of whether they are target analytes for a specific project, and the data user must be provided with the calibration verification data or a list of those analytes that exceeded the 15% limit. The effect of using the average of the response for all analytes for calibration verification would be similar to that for the initial calibration, namely that the quantitative results for those analytes where the difference is > 15% will include a greater uncertainty.

10.7 If any of the target analytes in the calibration verification fall outside of the retention time window established during initial calibration, the system is considered out of control. All samples analyzed after the last compliant calibration verification must be re-analyzed after instrument maintenance and re-calibration is successfully performed.

10.8 Instrument blanks must not contain any confirmed target analyte greater ½ of the reporting limit for the associated samples. Method blanks must not contain any confirmed target analyte greater than the reporting limit for the associated samples. If contamination is noted, stop all analyses, determine the source of the contamination, remove the source, and confirm the source is gone by reanalyzing an instrument blank and/or method blank as appropriate. QC and associated samples will be re-extracted and reanalyzed if the blank contamination is present > PQL in the samples.

11.0 WASTE DISPOSAL/POLLUTION PREVENTION

See the Standard Operating Procedure for "*Hazardous Waste Management Plan*," SWL-GA-114.

12.0 METHOD PERFORMANCE

12.1 The MDL concentrations are updated annually and are obtained using organic-free reagent water and sandy loam soil. MDL results are maintained in SWLO's "Red book" by the QA Department. Details for determining MDLs are given in Chapter One of SW-846 and the SOP for the "*Definition and Determination of Detection Limits*", SWL-GA-113. The MDL actually achievable in a given analysis will vary depending on detector response characteristics, irreducible noise from instrument electronics, and matrix effects.

12.2 The accuracy and precision obtainable following this method will be determined by the sample matrix, sample preparation technique, optional cleanup techniques, and calibration procedures used.

12.3 The laboratory should maintain control charts of LCS, MS, and MSD recoveries and relative percent deviations between MS and MSD recovery for accuracy and precision data. Surrogate recovery is also plotted. The control charts are monitored monthly for LCS and quarterly for MS and are maintained on file in the laboratory. Control limits are updated on an annual basis (See SOP "*Control Charts*", SWL-GA-112 for more details). See "Blue Book" for SWLO's most current control limits.

12.4 Examples of the separations on a pesticide column are shown in Attachments 2-5. QLs are calculated by multiplying the MDL value for an analyte by a number ranging from 5-10. The QL will, in no instance, be less than the MDL. See "Red Book" for SWLO's QLs.

13.0 REFERENCES

U.S.EPA SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods: "Method 3500B. Organic Extraction and Sample Preparation" Revision 2, December 1996.

U.S.EPA SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods: "Method 3510C. Separatory Liquid-Liquid Extraction" Revision 3, December 1996.

U.S.EPA SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods: "Method 3520C. Continuous Liquid-Liquid Extraction" Revision 3, December 1996.

U.S.EPA SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods: "Method 3540C. Soxhlet Extraction" Revision 3, December 1996.

U.S.EPA SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods: "Method 3550B. Sonication Extraction" Revision 2, December 1996.

U.S.EPA SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods: "Method 3580A. Waste Dilution" Revision 1, July 1992.

U.S.EPA SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods: "Method 8000B. Determinative Chromatographic Separations" Revision 2, December 1996.

U.S.EPA SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods: "Method 8081A. Organochlorine Pesticides by Gas Chromatography" Revision 1, December 1996.

Standard Operating Procedure, "Preventative Maintenance of Hewlett-Packard and Fisons Gas Chromatographs", SWL-OP-201.

Standard Operating Procedure, "Standards Receipt, Traceability, and Preparation", SWL-OP-202.

Standard Operating Procedure, "Laboratory Safety Plan", SWL-GA-111.

Standard Operating Procedure, "Control Charts", SWL-GA-112.

Standard Operating Procedure, "Definition and Determination of Detection Limits", SWL-GA-113.

Standard Operating Procedure, "Hazardous Waste Management Plan", SWL-GA-114.

Headquarters Air Force Center for Environmental Excellence Quality Assurance Project Plan, Version 2.0, January 1997.

U.S.EPA, Statement of Work OLM03.2, "Organic Analysis: Multi-media, Multi-concentration".

14.0 DEFINITIONS

PQL:..... Practical Quantitation Limit.

N/A:..... Not applicable

Material Safety Data Sheet (MSDS): .... Information provided by commercial vendors for the chemical products prepared by their company. The information on the MSDS includes but is not limited to composition, physical properties, hazard identification, first aid measures, emergency cleanup and/or response, exposure, and storage.

SDG: ..... The Sample Delivery Group; a unit within a single case that is used to identify a group of samples for delivery. A SDG is a group of 20 or fewer field samples within a case, received over a period of up to 14 calendar days (7 calendar days for 14-day turns). Data from all samples in a SDG are due concurrently.

15.0 ATTACHMENTS

15.1 Attachment 1: Sample Extraction Log

15.2 Attachment 2: Pesticide Chromatogram 1: INDA

15.3 Attachment 3: Pesticide Chromatogram 2: INDB

15.4 Attachment 4: Pesticide Chromatogram 3: INDAB

15.5 Attachment 5: Pesticide Chromatogram 4: TOXAPHENE

15.6 Attachment 6: Pesticide Chromatogram 5: TECHNICAL CHLORDANE

ATTACHMENT 1

Sample Extraction Log

[redacted] ME | [redacted] | | RUSH | | REEXTRACT | | TCLP

SAMPLE EXTRACTION LOG

SWLO
SOUTHWEST LABORATORY OF OKLAHOMA, INC

	SV		B	OTHER
T EX				
F		XXXXX		
KER				
C.				
ILE				

BATCH: 970617WE

EXTRACT DATE: 1997-06-17 11:57

LE ID / DOE #	CLIENT / CASE	DATE RECEIVED / CLIENT SAMPLE ID	MATRIX/LEVEL	SAMPLE pH	% MOISTURE	AMOUNT EXTRACTED (ml)	EXTRACTED VOLUME (ml)	GPC VOLUME (ml)	GPC ELUTE VOL (ml)	FLORISIL VOL CONC (ml)	FLORISIL FIN VOL CONC (ml)	VOL SV (ml)	FIN PROCESS VOLUME (ml)	SCREEN Y/N	MS SV (µl)	SURR SV (µl)	BASE pH CONFIRMATION	ACID pH CONFIRMATION	TCF ... (Y/N)
617WE			W			1000	250					10.0	1.0	N		1000			N
617WE			W			1000	250					10.0	1.0	N		1000			N
617WE			W			1000	250					10.0	1.0	N	1000	1000			N
617WE			W			1000	250					10.0	1.0	N	1000	1000			N
4.01 7	AATS-E 25501	1997-06-12 15:15 X6236 (CASE#25501)	W			1000	250					10.0	1.0	N		1000	·2	·2	N
4.02 7	AATS-E 25501	1997-06-12 15:15 X6237 (CASE#25501)	W			1000	250					10.0	1.0	N		1000	·2	·2	N
5.01 7	XXXXXXXX XXXXXXXX	XXXXXXXXXXXXXXXX XXXXXXXXXXXXXXXXXXXX	W			1000	250					10.0	1.0	N		1000	·2	·2	N
5.02 7	XXXXXXXX XXXXXXXX	XXXXXXXXXXXXXXXX XXXXXXXXXXXXXXXXXXXX	W			1000	250					10.0	1.0	N		1000	·2	·2	N

INITIALS

ING:	BL	EXTRACTION:	BL
UP:		RD:	BL
OWN:	BL	TV:	
O SPIKER:	BL	VERIFIER:	
SPIKER:	BL	VERIFIER:	
TED BY:	BL	REVIEWED BY:	

ID NUMBERS

FLORISIL CARTRIDGE #:

GPC COLUMN #:

SURROGATES ID CONC #:

MATRIX SPIKE SOLN/CONC #(s):

METHYLENE CHLORIDE LOT #:

SODIUM SULFATE LOT #:

COMMENTS

ATTACHMENT 2

Pesticide Chromatogram 1: INDA

Quantitation Report

Signal #1 : F:\HPCHEM\HP\1\DATA\05_03_00\1_22868.D\ECD1A.CH Vial: 19
Signal #2 : F:\HPCHEM\HP\1\DATA\05_03_00\1_22868.D\ECD2B.CH
Acq On : 03 May 2000 19:15 Operator: Jeff
Sample : INDAL31A Inst : HP_01
Misc : 5-346-4 Dilution: 1.00
IntFile Signal #1: events.e IntFile Signal #2: events2.e
Quant Time: May 4 9:58 2000 Quant Results File: 80E03.RES

Method : F:\HPCHEM\HP\1\METHODS\80E03.M
Title-Path : SW-846, METHOD 8080 - F:\HPCHEM\HP\1\DATA\05_01_00\
Last Update : Fri May 05 06:52:53 2000
Response via : Initial Calibration

Volume Inj. : 0.5uL
Signal #1 Phase : DB-17 Signal #2 Phase: DB-1701
Signal #1 Info : .32 Signal #2 Info : .32
Signal #1 Inst : HP_01A Signal #2 Inst : HP_01B

		Compound	RT#1	RT#2	Resp#1	Resp#2	ng/mL	ng/mL
		System Monitoring Compounds:						
1)	S	TCX	6.67	6.56	233110	124535	15.3	14.7
22)	S	DCB	18.86	17.41	517867	299152	29.1	30.5
		Target Compounds:						
2)	AE	alpha-BHC	8.07	8.33	246460	105277	13.6	12.5
3)	MA	gamma-BHC (Lindane	8.83	9.07	235281	105811	13.9	13.1
	MA	Heptachlor	9.44	9.45	268634	137248	14.5	14.1
	MA	Aldrin	0.00	0.00	0	0	N.D.	N.D.
	AE	beta-BHC	0.00	0.00	0	0	N.D.	N.D.
7)	A	delta-BHC	0.00	0.00	0	0	N.D.	N.D.
8)	A	Heptachlor Epoxide	0.00	0.00	0	0	N.D.	N.D.
9)	A	Endosulfan I	11.70	11.60	214797	108833	12.8	14.2
10)	A	gamma-Chlordane	0.00	0.00	0	0	N.D.	N.D.
11)	A	alpha-Chlordane	0.00	0.00	0	0	N.D.	N.D.
12)	A	4,4'-DDE	0.00	0.00	0	0	N.D.	N.D.
13)	MA	Dieldrin	12.24	12.22	410328	218228	28.4	27.4
14)	MA	Endrin	12.88	12.57	284432	146827	25.0	23.4
15)	A	Endosulfan II	0.00	0.00	0	0	N.D.	N.D.
16)	A	4,4'-DDD	13.04	13.23	290246	152441	28.0	25.8
17)	MA	4,4'-DDT	13.55	13.51	274052	147785	29.5	30.0
18)	A	Endrin Aldehyde	0.00	0.00	0	0	N.D. d	N.D.
19)	A	Endosulfan Sulfate	0.00	0.00	0	0	N.D.	N.D.
20)	AE	Methoxychlor	15.20	14.54	753810	485485	150.0	148.9
21)	A	Endrin Ketone	0.00	0.00	0	0	N.D. d	N.D. d

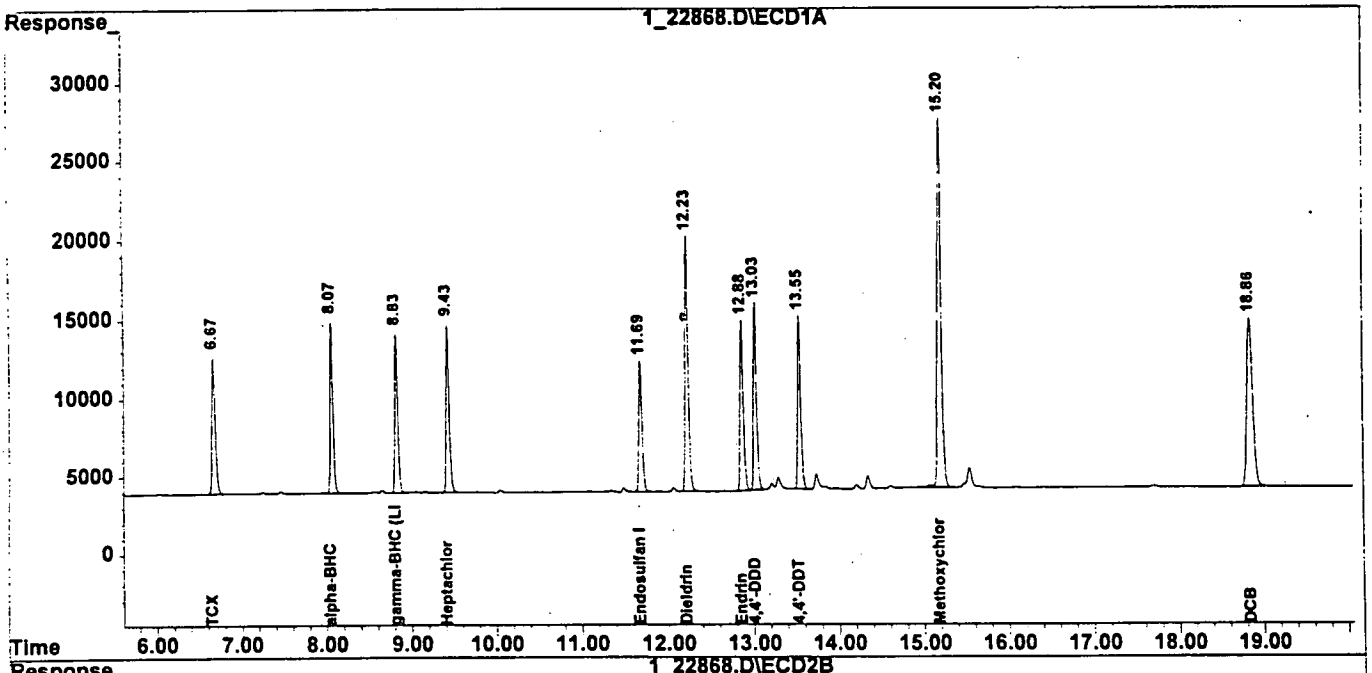
(f)=RT Delta > 1/2 Window (#)=Amounts differ by > 25% (m)=manual int.
(E)= > Highest cal. std. (d)=compound deleted (H) Dil. < med. std

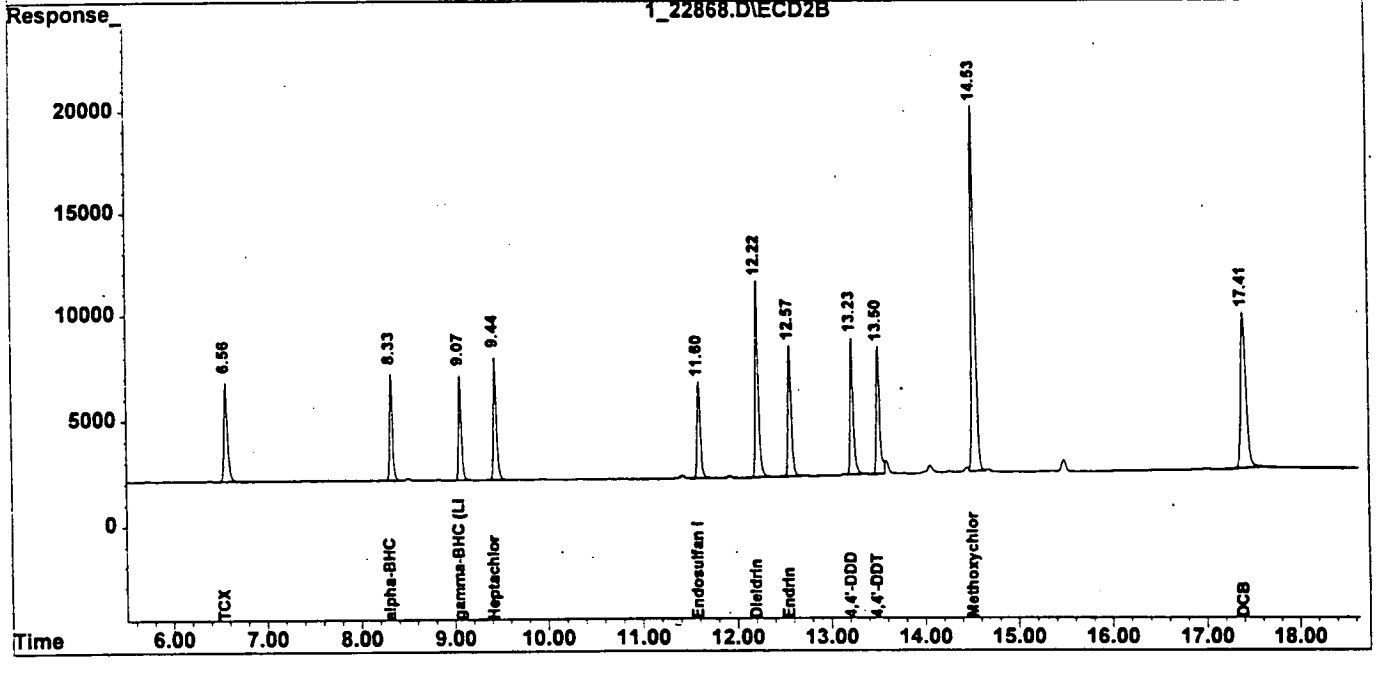
Chromatographic Report

Signal #1 : F:\HPCHEM\HP\1\DATA\05_03_00\1_22868.D\ECD1A.CH Vial: 19
Signal #2 : F:\HPCHEM\HP\1\DATA\05_03_00\1_22868.D\ECD2B.CH
Acq On : 03 May 2000 19:15 Operator: Jeff
Sample : INDAL31A Inst : HP_01
Misc : 5-346-4 Dilution: 1.00
IntFile Signal #1: events.e IntFile Signal #2: events2.e
Quant Time: May 4 9:58 2000 Quant Results File: 80E03.RES

Method : F:\HPCHEM\HP\1\METHODS\80E03.M
Title-Path : SW-846, METHOD 8080 - F:\HPCHEM\HP\1\DATA\05_01_00\
Last Update : Fri May 05 06:52:53 2000
Response via : Multiple Level Calibration

Volume Inj. : 0.5uL
Signal #1 Phase : DB-17 Signal #2 Phase: DB-1701
Signal #1 Info : .32 Signal #2 Info : .32
Signal #1 Inst : HP_01A Signal #2 Inst : HP_01B





ATTACHMENT 3

Pesticide Chromatogram 2: INDB

Quantitation Report

Signal #1 : F:\HPCHEM\HP\1\DATA\05_03_00\1_22869.D\ECD1A.CH Vial: 20
Signal #2 : F:\HPCHEM\HP\1\DATA\05_03_00\1_22869.D\ECD2B.CH
Acq On : 03-May 2000 19:40 Operator: Jeff
Sample : INDBL31A Inst : HP_01
Misc : 5-347-4 Dilution: 1.00
IntFile Signal #1: events.e IntFile Signal #2: events2.e
Quant Time: May 4 9:58 2000 Quant Results File: 80E03.RES

Method : F:\HPCHEM\HP\1\METHODS\80E03.M
Title-Path : SW-846, METHOD 8080 - F:\HPCHEM\HP\1\DATA\05_01_00\
Last Update : Fri May 05 06:52:53 2000
Response via : Initial Calibration

Volume Inj. : 0.5uL
Signal #1 Phase : DB-17 Signal #2 Phase: DB-1701
Signal #1 Info : .32 Signal #2 Info : .32
Signal #1 Inst : HP_01A Signal #2 Inst : HP_01B

		Compound	RT#1	RT#2	Resp#1	Resp#2	ng/mL	ng/mL
		System Monitoring Compounds:						
1)	S	TCX	6.67	6.56	221766	118585	14.5	14.0
22)	S	DCB	18.86	17.41	471242	268132	26.5	27.4
		Target Compounds:						
2)	AE	alpha-BHC	0.00	0.00	0	0	N.D.	N.D.
3)	MA	gamma-BHC (Lindane	0.00	0.00	0	0	N.D.	N.D.
	MA	Heptachlor	0.00	0.00	0	0	N.D.	N.D.
	MA	Aldrin	10.05	9.95	218374	107206	12.8	12.0
	AE	beta-BHC	8.97	10.39	130593	67401	12.9	12.6
7)	A	delta-BHC	9.66	10.82	183312	75740	12.6	11.7
8)	A	Heptachlor Epoxide	11.07	11.13	227747	111866	13.6	13.1
9)	A	Endosulfan I	0.00	0.00	0	0	N.D.	N.D.
10)	A	gamma-Chlordane	11.36	11.68	239104	123794	13.7	13.1
11)	A	alpha-Chlordane	11.64	11.77	241682	125686	15.4	13.0
12)	A	4,4'-DDE	12.08	11.92	359263	196965	26.9	25.5
13)	MA	Dieldrin	0.00	0.00	0	0	N.D.	N.D.
14)	MA	Endrin	0.00	0.00	0	0	N.D.	N.D.
15)	A	Endosulfan II	13.19	13.37	372008	187773	27.8	26.6
16)	A	4,4'-DDD	0.00	0.00	0	0	N.D.	N.D.
17)	MA	4,4'-DDT	0.00	0.00	0	0	N.D.	N.D.
18)	A	Endrin Aldehyde	13.75	14.06	256587	134275	23.7	23.9
19)	A	Endosulfan Sulfate	13.96	14.62	321649	168622	27.3	24.6
20)	AE	Methoxychlor	0.00	0.00	0	0	N.D.	N.D.
21)	A	Endrin Ketone	15.55	15.49	350895	163505	26.2	25.2

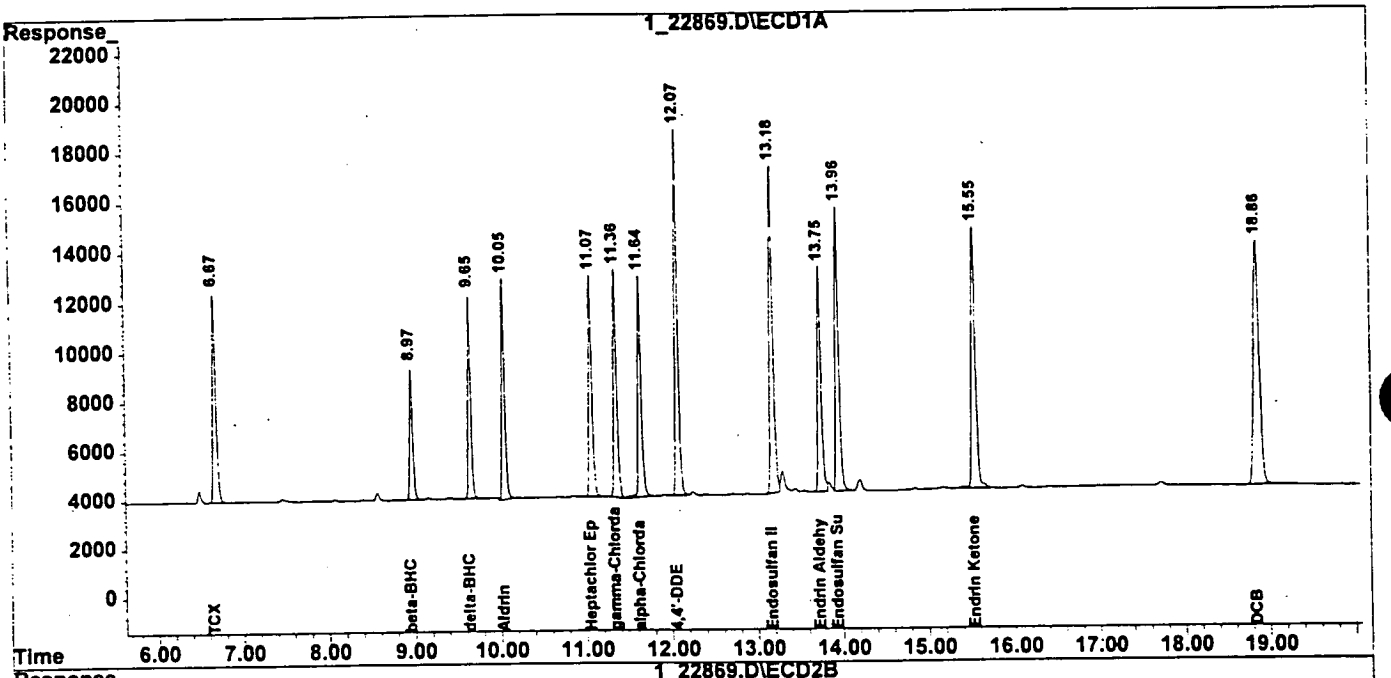
(f)=RT Delta > 1/2 Window (#)=Amounts differ by > 25% (m)=manual int.
(E)= > Highest cal. std. (d)=compound deleted (H) Dil. < med. std

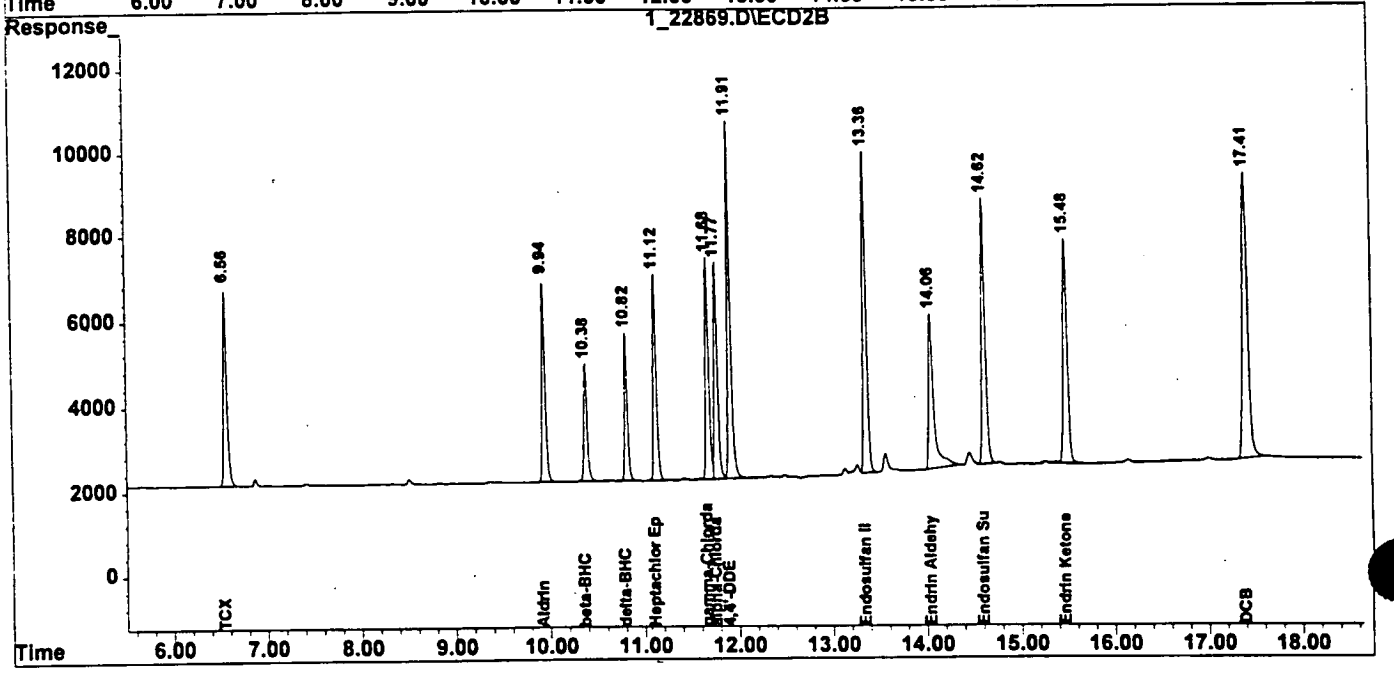
Chromatographic Report

Signal #1 : F:\HPCHEM\HP\1\DATA\05_03_00\1_22869.D\ECD1A.CH Vial: 20
Signal #2 : F:\HPCHEM\HP\1\DATA\05_03_00\1_22869.D\ECD2B.CH
Acq On : 03 May 2000 19:40 Operator: Jeff
Sample : INDBL31A Inst : HP_01
Misc : 5-347-4 Dilution: 1.00
IntFile Signal #1: events.e IntFile Signal #2: events2.e
Quant Time: May 4 9:58 2000 Quant Results File: 80E03.RES

Method : F:\HPCHEM\HP\1\METHODS\80E03.M
Title-Path : SW-846, METHOD 8080 - F:\HPCHEM\HP\1\DATA\05_01_00\
Last Update : Fri May 05 06:52:53 2000
Response via : Multiple Level Calibration

Volume Inj. : 0.5uL
Signal #1 Phase : DB-17 Signal #2 Phase: DB-1701
Signal #1 Info : .32 Signal #2 Info : .32
Signal #1 Inst : HP_01A Signal #2 Inst : HP_01B





ATTACHMENT 4

Pesticide Chromatogram 3: INDAB

Quantitation Report

Signal #1 : F:\HPCHEM\HP\1\DATA\05_03_00\1_22865.D\ECD1A.CH Vial: 16
Signal #2 : F:\HPCHEM\HP\1\DATA\05_03_00\1_22865.D\ECD2B.CH
Acq On : 03 May 2000 18:00 Operator: Jeff
Sample : INDABL31A Inst : HP_01
Misc : 5-379-4 Dilution: 1.00
IntFile Signal #1: events.e IntFile Signal #2: events2.e
Quant Time: May 4 7:45 2000 Quant Results File: 80E03.RES

Method : F:\HPCHEM\HP\1\METHODS\80E03.M
Title-Path : SW-846, METHOD 8080 - F:\HPCHEM\HP\1\DATA\05_03_00\
Last Update : Fri May 05 06:52:53 2000
Response via : Initial Calibration

Volume Inj. : 0.5uL
Signal #1 Phase : DB-17 Signal #2 Phase: DB-1701
Signal #1 Info : .32 Signal #2 Info : .32
Signal #1 Inst : HP_01A Signal #2 Inst : HP_01B

	Compound	RT#1	RT#2	Resp#1	Resp#2	ng/mL	ng/mL
	System Monitoring Compounds:						
1)	S TCX	6.67	6.56	242093	134014	15.9	15.8
22)	S DCB	18.86	17.41	282111	154284	15.9	15.7
	Target Compounds:						
2)	AE alpha-BHC	8.07	8.33	292499	129479	16.1	15.4
3)	MA gamma-BHC (Lindane	8.83	9.07	278010	127059	16.5	15.7
4)	MA Heptachlor	9.43	9.44	300098	157496	16.2	16.2
	MA Aldrin	10.04	9.94	273158	138636	16.0	15.5
[illegible]	AE beta-BHC	8.96	10.38	164883	85984	16.4	16.1
7)	A delta-BHC	9.65	10.82	232976	98526	16.0	15.2
8)	A Heptachlor Epoxide	11.06	11.12	270052	135088	16.1	15.8
9)	A Endosulfan I	11.69	11.60	270013	121202	16.0	15.8
10)	A gamma-Chlordane	11.35	11.68	278872	149975	16.0	15.8
11)	A alpha-Chlordane	11.64	11.77	246230	153137	15.6	15.9
12)	A 4,4'-DDE	12.07	11.91	435177	244612	32.6	31.6
13)	MA Dieldrin	12.23	12.22	468210	252573	32.5	31.7
14)	MA Endrin	12.87	12.57	376001	199678	33.1	31.8
15)	A Endosulfan II	13.18	13.36	422861	223393	32.0	31.7
16)	A 4,4'-DDD	13.03	13.23	334045	181775	32.4	30.8
17)	MA 4,4'-DDT	13.55	13.50	309066	171479	33.4	34.8
18)	A Endrin Aldehyde	13.74	14.05	344992	175263	31.6	31.2
19)	A Endosulfan Sulfate	13.95	14.62	373300	212610	31.8	31.0
20)	AE Methoxychlor	15.20	14.53	825514	532344	165.0	163.3
21)	A Endrin Ketone	15.55	15.48	439751	204345	32.8	31.5

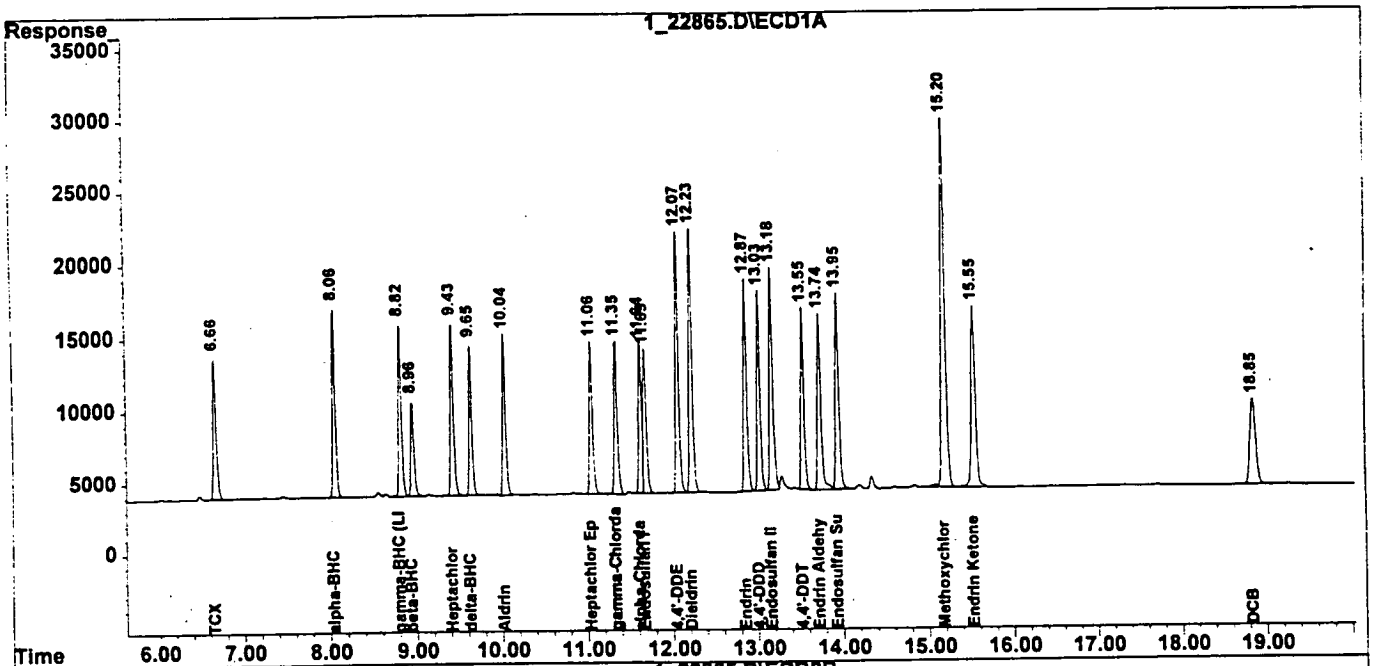
(f)=RT Delta > 1/2 Window (#)=Amounts differ by > 25% (m)=manual int.
(E)= > Highest cal. std. (d)=compound deleted (H) Dil. < med. std

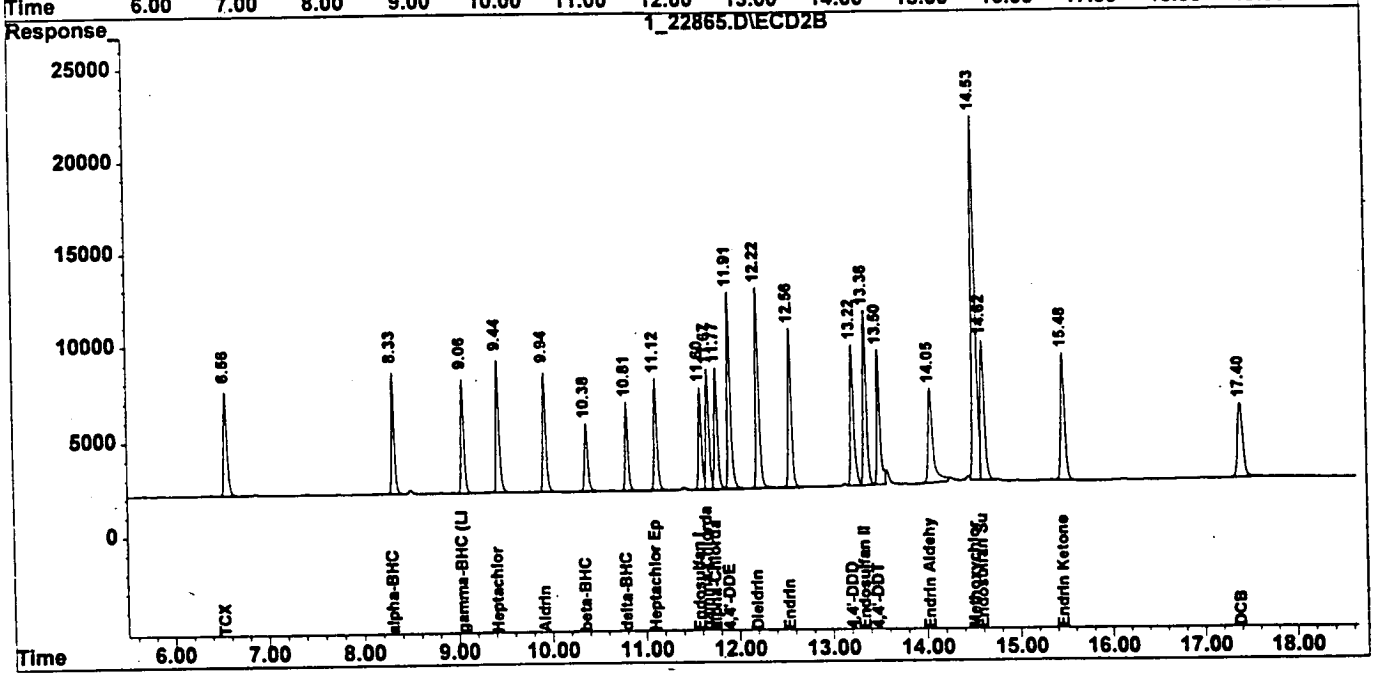
Chromatographic Report

Signal #1 : F:\HPCHEM\HP\1\DATA\05_03_00\1_22865.D\ECD1A.CH Vial: 16
Signal #2 : F:\HPCHEM\HP\1\DATA\05_03_00\1_22865.D\ECD2B.CH
Acq On : 03 May 2000 18:00 Operator: Jeff
Sample : INDABL31A Inst : HP_01
Misc : 5-379-4 Dilution: 1.00
IntFile Signal #1: events.e IntFile Signal #2: events2.e
Quant Time: May 4 7:45 2000 Quant Results File: 80E03.RES

Method : F:\HPCHEM\HP\1\METHODS\80E03.M
Title-Path : SW-846, METHOD 8080 - F:\HPCHEM\HP\1\DATA\05_03_00\
Last Update : Fri May 05 06:52:53 2000
Response via : Multiple Level Calibration

Volume Inj. : 0.5uL
Signal #1 Phase : DB-17 Signal #2 Phase: DB-1701
Signal #1 Info : .32 Signal #2 Info : .32
Signal #1 Inst : HP_01A Signal #2 Inst : HP_01B





ATTACHMENT 5

Pesticide Chromatogram 4: TOXAPHENE

Quantitation Report

Signal #1 : F:\HPCHEM\HP\1\DATA\05_03_00\1_22862.D\ECD1A.CH Vial: 13
Signal #2 : F:\HPCHEM\HP\1\DATA\05_03_00\1_22862.D\ECD2B.CH
Acq On : 03 May 2000 16:44 Operator: Jeff
Sample : TOXAPHL11A Inst : HP_01
Misc : 5-298-4 Dilution: 1.00
IntFile Signal #1: events.e IntFile Signal #2: events2.e
Quant Time: May 4 7:56 2000 Quant Results File: MULTIE03.RES

Method : F:\HPCHEM\HP\1\METHODS\MULTIE03.M
Title-Path : PCB/TOXAPHENE - F:\HPCHEM\HP\1\DATA\05_03_00\
Last Update : Tue Apr 04 08:59:35 2000
Response via : Initial Calibration

Volume Inj. : 0.5uL
Signal #1 Phase : DB-17 Signal #2 Phase: DB-1701
Signal #1 Info : 0.32mm Signal #2 Info : 0.32mm
Signal #1 Inst : HP_01A Signal #2 Inst : HP_01B

	Compound	RT#1	RT#2	Resp#1	Resp#2	ng/mL	ng/mL
	System Monitoring Compounds:						
1)	S TCX	6.66	6.56	96517	48756	5.385	5.061
10)	S DCB	18.85	17.40	147471	76627	6.537	6.563
	Target Compounds:						
6)	L8 Toxaphene	12.46	13.57	100185	63149	437.5	466.2
7)	L8 Toxaphene {2}	12.58	13.84	88851	87055	387.8	446.3
	L8 Toxaphene {3}	14.81	14.64	198452	53431	322.5	350.5
	L8 Toxaphene {4}	15.50	15.00	98921	50124	327.8	297.2
	Sum Toxaphene			486409	253759	1475.6	1560.1
	Average Toxaphene					368.897	390.031

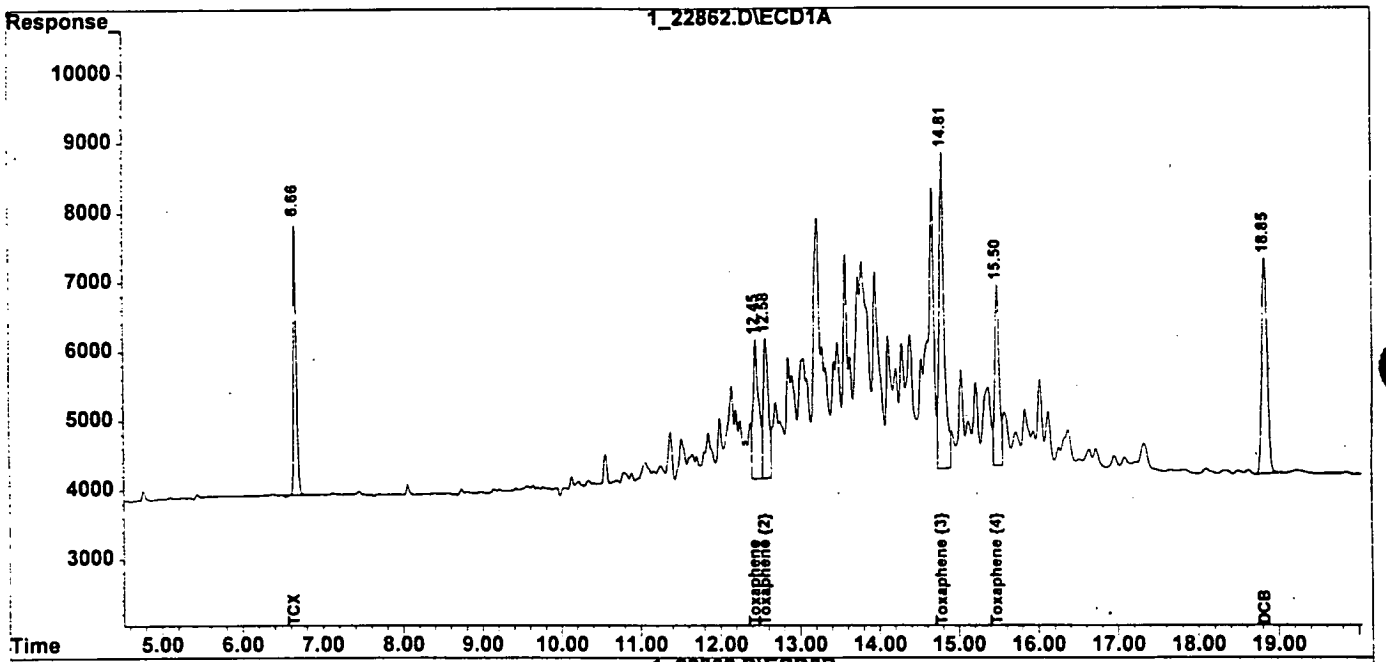
(f)=RT Delta > 1/2 Window (#)=Amounts differ by > 25% (m)=manual int.
(E)= > Highest cal. std. (d)=compound deleted (H) Dil. < med. std

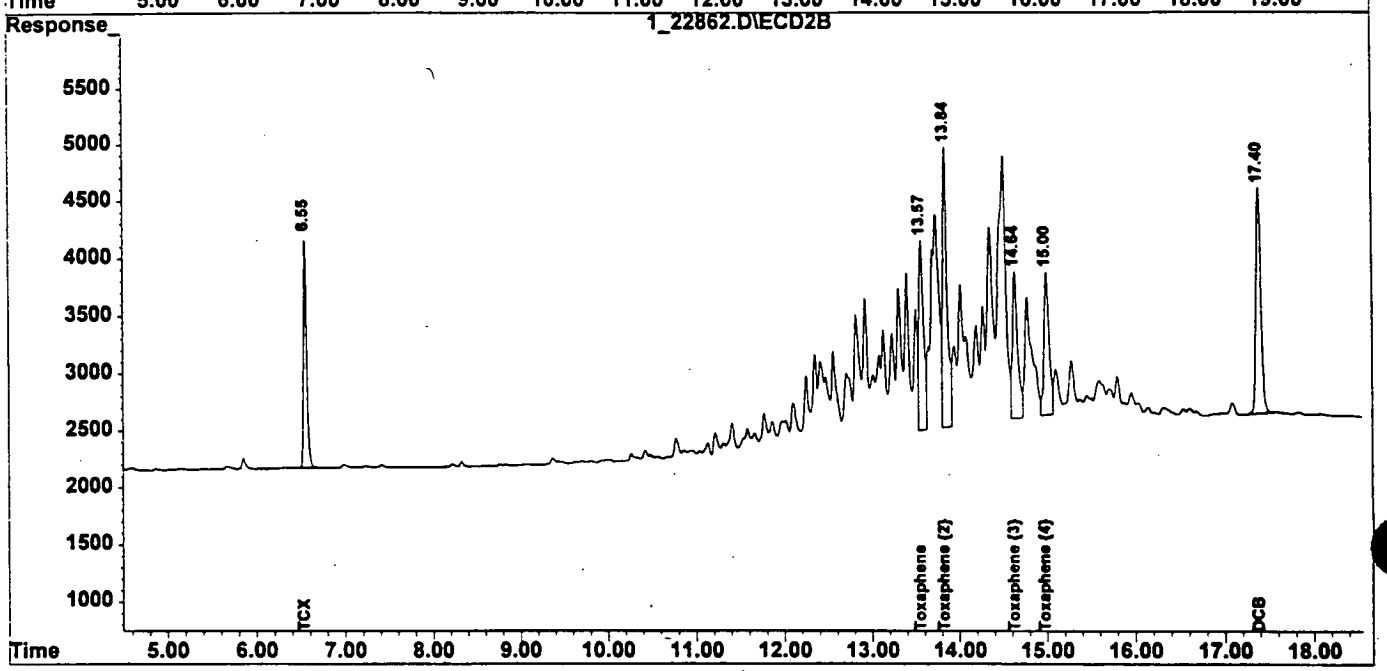
Chromatographic Report

Signal #1 : F:\HPCHEM\HP\1\DATA\05_03_00\1_22862.D\ECD1A.CH Vial: 13
Signal #2 : F:\HPCHEM\HP\1\DATA\05_03_00\1_22862.D\ECD2B.CH
Acq On : 03 May 2000 16:44 Operator: Jeff
Sample : TOXAPHL11A Inst : HP_01
Misc : 5-298-4 Dilution: 1.00
IntFile Signal #1: events.e IntFile Signal #2: events2.e
Quant Time: May 4 7:56 2000 Quant Results File: MULTIE03.RES

Method : F:\HPCHEM\HP\1\METHODS\MULTIE03.M
Title-Path : PCB/TOXAPHENE - F:\HPCHEM\HP\1\DATA\05_03_00\
Last Update : Tue Apr 04 08:59:35 2000
Response via : Single Level Calibration

Volume Inj. : 0.5uL
Signal #1 Phase : DB-17 Signal #2 Phase: DB-1701
Signal #1 Info : 0.32mm Signal #2 Info : 0.32mm
Signal #1 Inst : HP_01A Signal #2 Inst : HP_01B





ATTACHMENT 6

Pesticide Chromatogram 5: TECHNICAL CHLORDANE

Quantitation Report

Signal #1 : F:\HPCHEM\HP\1\DATA\05_03_00\1_22861.D\ECD1A.CH Vial: 12
Signal #2 : F:\HPCHEM\HP\1\DATA\05_03_00\1_22861.D\ECD2B.CH
Acq On : 03 May 2000 16:19 Operator: Jeff
Sample : TCHLORL11A Inst : HP_01
Misc : 5-362-4 Dilution: 1.00
IntFile Signal #1: events.e IntFile Signal #2: events2.e
Quant Time: May 4 7:56 2000 Quant Results File: MULTIE03.RES

Method : F:\HPCHEM\HP\1\METHODS\MULTIE03.M
Title-Path : PCB/TOXAPHENE - F:\HPCHEM\HP\1\DATA\05_01_00\
Last Update : Tue Apr 04 08:59:35 2000
Response via : Initial Calibration

Volume Inj. : 0.5uL
Signal #1 Phase : DB-17 Signal #2 Phase: DB-1701
Signal #1 Info : 0.32mm Signal #2 Info : 0.32mm
Signal #1 Inst : HP_01A Signal #2 Inst : HP_01B

	Compound	RT#1	RT#2	Resp#1	Resp#2	ng/mL	ng/mL
	System Monitoring Compounds:						
1)	S TCX	6.67	6.56	89623	48167	4.643	4.940
10)	S DCB	18.85	17.40	112801	58374	3.825	3.809
	Target Compounds:						
2)	L9 T. Chlordane	9.14	9.16	10126	3392	10.2	9.825
3)	L9 T. Chlordane {2}	9.43	9.44	13161	6671	9.848	10.3
	L9 T. Chlordane {3}	11.35	11.68	21528	9136	8.798	9.247
	L9 T. Chlordane {4}	11.64	11.77	18456	13683	11.0	9.362
	Sum T. Chlordane			63270	32881	39.8	38.7
	Average T. Chlordane					9.959	9.676

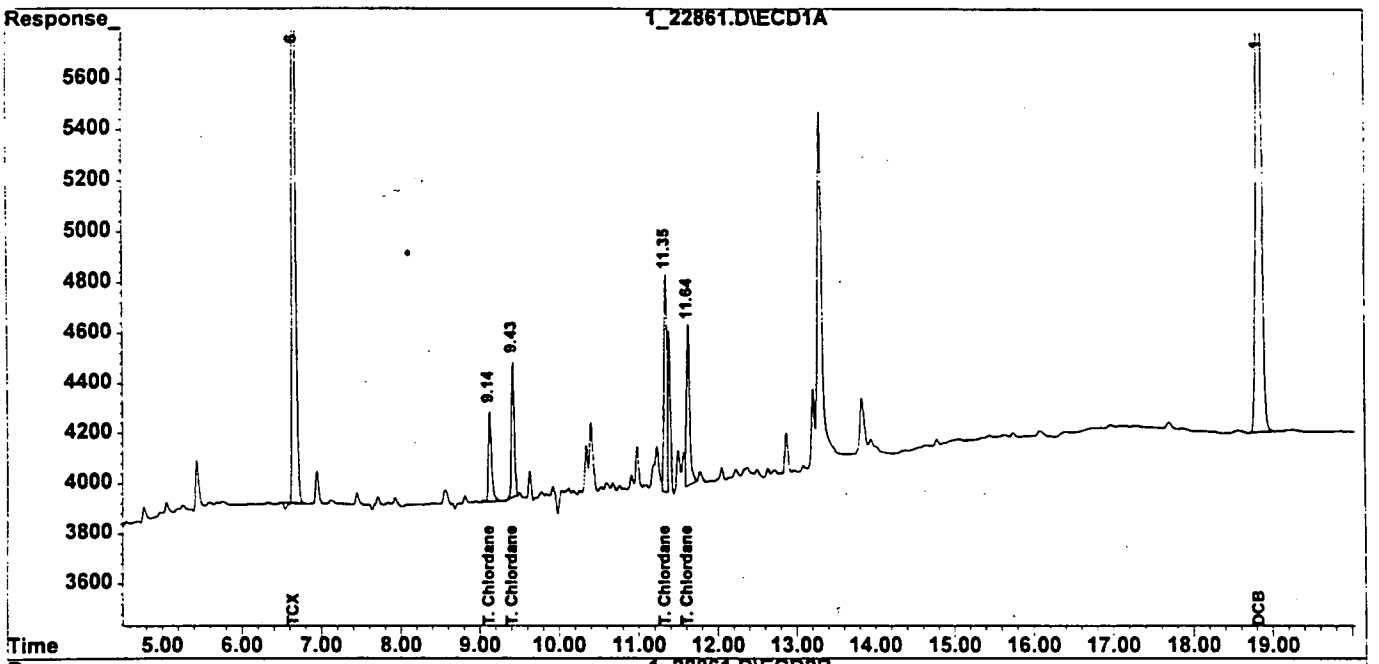
(f)=RT Delta > 1/2 Window (#)=Amounts differ by > 25% (m)=manual int.
(E)= > Highest cal. std. (d)=compound deleted (H) Dil. < med. std

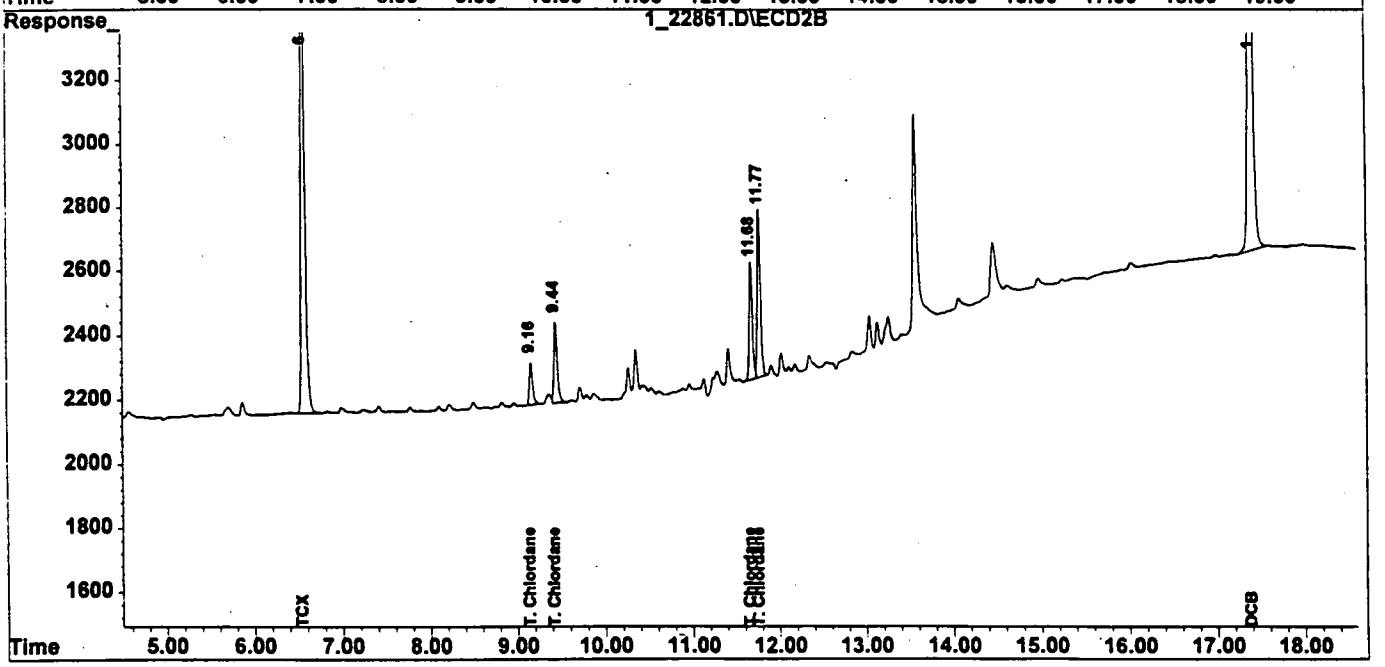
Chromatographic Report

Signal #1 : F:\HPCHEM\HP\1\DATA\05_03_00\1_22861.D\ECD1A.CH Vial: 12
Signal #2 : F:\HPCHEM\HP\1\DATA\05_03_00\1_22861.D\ECD2B.CH
Acq On : 03 May 2000 16:19 Operator: Jeff
Sample : TCHLORL11A Inst : HP_01
Misc : 5-362-4 Dilution: 1.00
IntFile Signal #1: events.e IntFile Signal #2: events2.e
Quant Time: May 4 7:56 2000 Quant Results File: MULTIE03.RES

Method : F:\HPCHEM\HP\1\METHODS\MULTIE03.M
Title-Path : PCB/TOXAPHENE - F:\HPCHEM\HP\1\DATA\05_01_00\
Last Update : Tue Apr 04 08:59:35 2000
Response via : Single Level Calibration

Volume Inj. : 0.5uL
Signal #1 Phase : DB-17 Signal #2 Phase: DB-1701
Signal #1 Info : 0.32mm Signal #2 Info : 0.32mm
Signal #1 Inst : HP_01A Signal #2 Inst : HP_01B





SOUTHWEST LABORATORY OF OKLAHOMA, INC. and
AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.

TM

Standard Operating Procedure

Analysis of PCBs by GC, SW-846, Method 8082

Document No.: SWL-OP-415

Rev No. / Date: Rev. 3.1 — 02/07/00

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APPROVALS	DATES
Procedure Prepared By	2-8-00 Date
Organic Program Manager	3-13-00 Date
Laboratory Safety Officer	3/14/2000 Date
Laboratory QA/QC Officer	3/13/00 Date

(Effective Date is 5 calendar days after the last signature above - QA/QC Officer)

Document Status

[ ] Controlled DC No. _____ Issued to: _____ Date: _____ QA: _____

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ANALYSIS OF PCBs BY GC, SW-846, METHOD 8082

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1.0 SCOPE AND APPLICATION

1.1 This method is designed to determine the concentrations of various polychlorinated biphenyls (PCBs) as Aroclors™ or individual PCB congeners in extracts from solid and liquid matrices. The method is based on EPA SW-846 methods 8000B and 8082 for analysis and methods 3500B, 3510C, 3520C, 3540C, 3550B, 3580A, 3600C, 3660B, and 3665A for extraction and cleanup. The method can be used to determine analyte concentrations of PCBs in the parts per billion (ppb) range in water and soil matrices. Test codes encompassed by this method include GC601, 602, 610 and GC699.

1.2 A large number of compounds will give response in the electron capture detector (ECD) using this method. Tables 1, 2, and 3 represent the compounds that may be analyzed by this method.

TABLE 1
Aroclor Target Compound List

ANALYTE	CAS #
Aroclor 1016	12674-11-2
Aroclor 1221	11104-28-2
Aroclor 1232	11141-16-5
Aroclor 1242	53469-21-9
Aroclor 1248	12672-29-6
Aroclor 1254	11097-69-1
Aroclor 1260	11096-82-5

TABLE 2
PCB Congener Target Compound List

ANALYTE	CAS #	IUPAC#
2-Chlorobiphenyl	2051-60-7	1
2,3-Dichlorobiphenyl	16605-91-7	5
2,2',5-Trichlorbiphenyl	37680-65-2	18
2,4',5-Trichlorobiphenyl	16606-02-3	31
2,2'3,5'-Tetrachlorobiphenyl	41464-39-5	44
2,2',5,5'-Tetrachlorbiphenyl	35693-99-3	52
2,3',4,4'-Tetrachlorobiphenyl	32598-10-0	66
2,2',3,4,5'-Pentachlorobiphenyl	38380-02-8	87
2,2',4,5,5'-Pentachlorobiphenyl	37680-73-2	101
2,3,3',4',6-Pentachlorobiphenyl	38380-03-9	110
2,2',3,4,4',5'-Hexachlorobiphenyl	35065-28-2	138
2,2',3,4,5,5'-Hexachlorobiphenyl	52712-04-6	141
2,2',3,5,5',6-Hexachlorobiphenyl	52663-63-5	151
2,2'-4,4'-5,5'-Hexachlorobiphenyl	35065-27-1	153
2,2',3,3',4,4',5-Heptachlorobiphenyl	35065-30-6	170
2,2',3,4,4',5,5'-Heptachlorobiphenyl	35065-29-3	180
2,2',3,4,4',5',6-Heptachlorobiphenyl	52663-69-1	183
2,2',3,4',5,5',6-Heptachlorobiphenyl	52663-68-0	187
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	40186-72-9	206

Table 3
Extended List Aroclor Target Compounds

ANALYTE	CAS #
Aroclor 1262	37324-23-5
Aroclor 1268	11100-14-4

1.3 This method utilizes wide bore capillary columns (0.53 mm ID) or narrow bore capillary (0.32 or 0.25mm ID) columns for both the quantitation and confirmation analyses. There are no method deviations from those referenced in Section 13.0.

1.4 See the Redbook for most current quantitation limits (QLs).

2.0 METHOD SUMMARY

2.1 Continuous liquid-liquid or separatory funnel extraction procedures are employed for the extraction of aqueous samples. Sonication or Soxhlet extraction is required for soil/sediment samples. Both neat and diluted organic liquids may be analyzed by direct injection (Method 3580A, Waste Dilution). All samples go through sulfuric acid cleanup by method 3665.

Continuous liquid-liquid extraction is not a recommended sample preparation method for Method 8082 analyses. Chlorinated compounds may be de-chlorinated. Therefore, method 3510C is recommended for 8082 analyses. Method 3520C, however, may be used in certain instances if pre-approved by management.

2.2 Samples are routinely analyzed on 2 columns of different stationary phases simultaneously to expedite the confirmation of target analytes. All calibration criteria apply to both columns independently. The lower analytical result (between the 2 columns) is routinely reported unless there is apparent evidence of negative matrix interference.

2.3 Columns are calibrated using the external calibration procedure with 5 concentrations of the analytes covering the working linear range of the ECD detectors beginning from the level of quantitation or below. Calibration criteria specified in method 8000B are followed (unless otherwise specified).

2.4 Calibration verification standards are analyzed each 12-hour shift (not to exceed 20 samples) and before the start of an analysis sequence so that they bracket the samples.

2.5 A method blank, laboratory control spike, and a matrix spike/matrix spike duplicate must be prepared for all batches of 20 samples or less.

3.0 HEALTH AND SAFETY

3.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a

current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data Sheets (MSDS) is available to all personnel involved in chemical analysis.

3.2 PCBs have been tentatively classified as known or suspected, human or mammalian carcinogens. Primary standards of these toxic compounds must be prepared in a hood. A NIOSH/MESA approved toxic gas respirator must be worn when the analyst handles high concentrations of these toxic compounds.

3.3 Caution should be taken when working with all sample extracts and calibration standards. Samples are of unknown origin and should be considered hazardous. Many of the spiking solutions used, along with chlorinated solvents, are considered carcinogens. Concentrated acids and bases, if not handled properly, can cause severe burns.

3.4 Gloves should be used for the manipulation of all sample extracts and calibration standards and during the preparation of all acid and base solutions. Wear personal protective equipment (PPE) and goggles or face shield when warranted. See the standard operating procedure for "Laboratory Safety Plan", SWL-GA-111 for detailed safety procedures.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

4.1 Sample Extraction

4.1.1 Aqueous samples must be extracted within 7 days of collection. Solid samples must be extracted within 14 days of collection.

4.1.2 Extracts must be stored under refrigeration at 4°C (±2°C) and analyzed within 40 days of extraction.

4.2 Sample Storage

4.2.1 The samples must be protected from light and refrigerated at 4°C (±2°C) from the time of receipt until 60 days after delivery to the client of a complete, reconciled sample data package. After 60 days the samples may be disposed of in a manner that complies with all applicable regulations.

4.2.2 The sample must be stored in an atmosphere demonstrated to be free of all potential contaminants.

4.2.3 Samples, sample extracts, and standards must be stored separately.

4.3 Sample Extract Storage

4.3.1 Sample extracts must be protected from light and stored at 4°C (±2°C) until 365 days after delivery to the client of a complete data package.

4.3.2 Sample extracts must be stored in an atmosphere demonstrated to be free of all potential contaminants.

4.3.3 Samples, sample extracts, and standards must be stored separately.

4.4 All containers should be glass jars or bottles with Teflon lids. A minimum of one liter is required for aqueous samples. A minimum of 80g is required for soil samples (125-mL glass jar).

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

5.1 Sources of interference in this method can be grouped into three broad categories: contaminated solvents, reagents or sample processing hardware; contaminated GC carrier gas, parts, column surfaces or detector surfaces; and the presence of co-eluting compounds in the sample matrix to which the ECD will respond. Knowledge of good laboratory practices is assumed, including steps to be followed in routine testing and cleanup of solvents, reagents and sample processing hardware, and instrument maintenance. The discussion that follows focuses on sources of interference associated with the sample matrix and compound classes that represent common sources of interference, particularly phthalate esters, organosulfur compounds, lipids, and waxes. Interferences co-extracted from the samples will vary considerably from sample to sample. While general cleanup techniques are referenced or provided as part of this method, unique samples may require additional cleanup approaches to achieve desired degrees of discrimination and quantitation.

5.2 Interferences by phthalate esters introduced during sample preparation can pose a major problem in pesticide determinations. These materials may be removed prior to analysis using sulfuric acid/permanganate cleanup (3665). Common flexible plastics contain varying amounts of phthalate esters that are easily extracted or leached from such materials during laboratory operations. Cross-contamination of clean glassware routinely occurs when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled. Interferences from phthalate esters can best be minimized by avoiding contact with all plastic materials and checking all solvents and reagents for phthalate contamination. Exhaustive cleanup of solvents, reagents and glassware may be required to eliminate background phthalate ester contamination.

5.3 The presence of elemental sulfur will result in large peaks that interfere with the detection of later eluting organochlorine pesticides. Method 3660 is suggested for removal of sulfur.

5.4 Differing Aroclor mixtures will co-elute to some degree. Care must be taken to choose non-coeluting peaks for quantitation, (if possible).

5.5 If oven drying of glassware used for PCB analysis is employed, special care must be taken. PCBs are readily volatilized in the oven and spread to other glassware. Therefore, do not dry glassware from samples containing high concentrations of PCBs with glassware that may be used for trace analyses.

6.0 EQUIPMENT/APPARATUS

6.1 Gas chromatograph — Analytical system complete with gas chromatograph suitable for split-splitless injection and all required accessories including

syringes, analytical columns, gases, electron capture detector, and recorder/integrator or data system.

6.1.1 HP 5890 Gas Chromatograph with ECD detector(s) and a 7673 autosampler. The autosampler must be capable of 1 µL or 2 µL injections. The carrier gas should be ultra pure helium or hydrogen, and the makeup gas (detector gas) should be ultra pure nitrogen or 5% methane/95% argon.

The following column pairs are recommended:

Column pair 1 — DB-17MS, DB-XLB fused silica capillary columns, 0.25mm ID x 30m, 0.25um film thickness.

Column pair 2 — RTX CLPesticides, 30m x 0.32mm ID, 0.5um film thickness, or 15m x 0.25mm ID, 0.25 film thickness, RTX CLPesticides II, 30m x 0.32mm ID, 0.25um film thickness, or 15m x 0.25mm ID, 0.25um film thickness.

Column pair 3 — DB17/1701P, 30m x 0.32mm ID, 0.25um film thickness.

7.0 REAGENTS

7.1 Reagent or pesticide grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 All gases (i.e., carrier, auxiliary, etc.) should be at least high purity grade.

7.3 Standards preparation:

Prepare individual stock standard solutions in n-hexane or iso-octane at the following concentrations every six months; or sooner if the solution has degraded or concentrated (Assign log numbers to each of the solutions and log them into the Standards Log Book; see Standard Operating Procedure for "Standards Receipt, Traceability, and Preparation", SWL-OP-202).

7.3.1 Stock Standard Solutions - Individual solutions are obtained from AccuStandard or equivalent supplier at concentrations of 1000µg/mL in hexane.

7.3.1.1 Concentrated and certified standard mixtures can be obtained from several sources. For example, Restek and Supelco supply the Aroclor mixtures. It is recommended that these be used for traceability purposes. Stock standard solutions can then be diluted directly from the concentrates.

7.3.1.2 The surrogate standards tetrachloro-m-xylene and decachlorobiphenyl are added to all standards, samples and blanks.

7.3.1.3 Stock standard solutions must be kept refrigerated at 4°C (±2°C) for no longer than 6 months.

7.3.2 Prepare solutions of the calibration standards for Aroclors 1016/1260 and/or PCB congeners at 5 concentration levels beginning at the quantitation limit and spanning the linear working range of the detector. See Tables 4 and 5 for the solution concentrations. See Table 6 for the extra Aroclor compounds. Calibration standards must remade at least every 6 months, or verified against new standards before use.

Table 4
Aroclor Calibration Standards — Low Point Concentration ng/mL (Level 1)

Aroclor Mixtures	Low Point Concentration ng/mL (Level 1)
AR1016	100
AR1221	100
AR1232	100
AR1242	100
AR1248	100
AR1254	100
AR1260	100

Table 5
PCB Congener Mixture — Low Point Concentration ng/mL (Level 1)

PCB Congener Mixture	Low Point Concentration ng/mL (Level 1)
2-Chlorobiphenyl	10
2,3-Dichlorobiphenyl	2
2,2',5-Trichlorbiphenyl	2
2,4',5-Trichlorobiphenyl	2
2,2'3,5'-Tetrachlorobiphenyl	2
2,2',5,5'-Tetrachlorbiphenyl	2
2,3',4,4'-Tetrachlorobiphenyl	2
2,2',3,4,5'-Pentachlorobiphenyl	2
2,2',4,5,5'-Pentachlorobiphenyl	2
2,3,3',4',6-Pentachlorobiphenyl	2
2,2',3,4,4',5'-Hexachlorobiphenyl	2
2,2',3,4,5,5'-Hexachlorobiphenyl	2
2,2',3,5,5',6-Hexachlorobiphenyl	2
2,2'-4,4'-5,5'-Hexachlorobiphenyl	2
2,2',3,3',4,4',5-Heptachlorobiphenyl	2
2,2',3,4,4',5,5'-Heptachlorobiphenyl	2
2,2',3,4,4',5',6-Heptachlorobiphenyl	2
2,2',3,4',5,5',6-Heptachlorobiphenyl	2
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	2

Table 6
Extended List Mixture — Low Point Concentration ng/mL (Level 1)

Extended List Mixture	Low Point Concentration ng/mL (Level 1)
AR1262	100
AR1268	100

Prepare all solutions in hexane or isooctane. Keep stored at 4°C (±2°C) no longer than 6 months.

7.3.3 Spiking Solutions:

7.3.3.1 Surrogate Solution

7.3.3.1.1 The surrogates, Tetrachloro-m-xylene and Decachlorobiphenyl, are added to all standards, samples, matrix spikes, and blanks. Prepare a surrogate spiking solution of 0.2 µg/mL of each of the two compounds in acetone or methanol. The solution should be checked frequently for stability. The solution must be replaced after six months, or sooner, if comparison with quality control check-samples indicates a problem.

CAUTION: Analysts must allow all spiking solutions to equilibrate to room temperature before use.

7.3.3.2 Aroclor Matrix Spiking Solution

7.3.3.2.1 Prepare a spiking solution in methanol (acetone produces interfering aldol-condensation products) that contains the Aroclors or PCB congeners in the concentrations specified in Tables 7 and 8.

Table 7
Matrix Spiking Solution (µg/mL)

ANALYTE	Concentration (µg/mL)
Aroclor 1016	9.0
Aroclor 1260	9.0

Table 8
PCB Congener Matrix Spiking Solution (µg/mL)

ANALYTE	CONCENTRATION (µg/mL)
2-Chlorobiphenyl	0.40
2,3-Dichlorobiphenyl	0.08
2,2',5-Trichlorbiphenyl	0.08
2,4',5-Trichlorobiphenyl	0.08
2,2'3,5'-Tetrachlorobiphenyl	0.08
2,2',5,5'-Tetrachlorbiphenyl	0.08
2,3',4,4'-Tetrachlorobiphenyl	0.08
2,2',3,4,5'-Pentachlorobiphenyl	0.08
2,2',4,5,5'-Pentachlorobiphenyl	0.08
2,3,3',4',6-Pentachlorobiphenyl	0.08
2,2',3,4,4',5'-Hexachlorobiphenyl	0.08
2,2',3,4,5,5'-Hexachlorobiphenyl	0.08
2,2',3,5,5',6-Hexachlorobiphenyl	0.08
2,2'-4,4'-5,5'-Hexachlorobiphenyl	0.08
2,2',3,3',4,4',5-Heptachlorobiphenyl	0.08
2,2',3,4,4',5,5'-Heptachlorobiphenyl	0.08
2,2',3,4,4',5',6-Heptachlorobiphenyl	0.08
2,2',3,4',5,5',6-Heptachlorobiphenyl	0.08
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	0.08

7.3.3.2.2 The solution must be prepared every six months or sooner if the solution has degraded or concentrated.

8.0 PROCEDURE

8.1 Gas chromatography set points:

The following set points are recommended:

Column Conditions:

DB-17MS/XLB

Carrier Gas:	Hydrogen at 12psi
Injector Temp:	220
Detector Temp:	320
Initial Temp:	110, hold for 0.5 minute
Temperature Prog.:	110 to 150 at 25 degrees/minute
	150 to 250 at 9 degrees/minute
	250 to 320 at 25 degrees/minute
Final Temp:	320 hold for 3.0 minutes.

DB-17/1701

Carrier Gas:	Hydrogen at 7psi
Injector Temp:	220
Detector Temp:	320
Initial Temp:	110, hold for 0.5 minutes
Temperature Prog:	110 to 140 at 20 degrees/minute
	140 to 280 at 12 degrees/minute
Final Temp:	280 hold for 6.33 minutes

RTX-CLPesticides/CLPesticides II 30m x 0.32mm

Carrier Gas:	Hydrogen at 7psi
Injector Temp:	220
Detector Temp:	320
Initial Temp:	140, hold for 0.5 minutes
Temperature Prog:	140 to 250 at 9 degrees/minute
	250 to 300 at 15 degrees/minute
Final Temp:	300 hold for 4.44 minutes

RTX-CLPesticides/CLPesticides II 15m x 0.25mm

Carrier Gas:	Helium at 13 psi
Injector Temp:	220
Detector Temp:	320
Initial Temp:	110, hold for 0.5 minutes
Temperature Prog:	110 to 260 at 7.5 degrees/minute
	260 to 290 at 11 degrees/minute
Final Temp:	290 hold for 2.0 minutes

DB-17/1701

Carrier Gas:	Helium at 11 psi
Injector Temp:	220
Detector Temp:	320
Initial Temp:	110, hold for 0.5 minutes
Temperature Prog:	110 to 140 at 20 degrees/minute
	140 to 280 at 12 degrees/minute
Final Temp:	280 hold for 6.5 minutes

8.2 Calibration

8.2.1 Refer to Method 8000B for proper calibration techniques. Percent relative standard deviation must be ≤ 20 for all analytes over the five-point calibration range to assume linearity through the origin (and use the mean calibration factor for quantitation). In those instances where the RSD for one or more analytes exceeds 20%, the initial calibration may still be acceptable for mean calibration factor use if the following conditions are met: 1) The mean of the RSD values for all analytes in the calibration is ≤ 20%. The mean RSD is calculated by summing the RSD value for each analyte and dividing by the total number of analytes. If no analyte has an RSD above 20%, then the mean RSD calculation need not be performed. 2) The mean RSD criterion applies to all analytes in the standard, regardless of whether or not they are of interest for a specific project. In other words, if the target analyte is part of the calibration standard, its RSD value is included in the evaluation. 3) The data user must be provided with either a summary of the initial calibration data or a specific list of those compound for which the RSD exceeded 20% and the results of the mean RSD calculation.

8.2.2 The procedure of external calibration is used. Quantitation using the mean calibration factor is preferred, however, linear least squares and quadratic fit may be used. If a quadratic fit is to be used, six levels of standards must be analyzed for the initial calibration.

8.2.3 SWLO's policy is to use a five-point calibration for Aroclors 1016 and 1260 and a single point calibration for the other 5 Aroclors.

8.2.4 Because of the low concentration of PCB standards injected on a GC/ECD, column adsorption may be a problem when the GC has not been used for a day. Therefore, the GC column should be primed or deactivated by injecting a PCB standard mixture approximately 4 times more concentrated than the mid-concentration standard. Inject this standard mixture plus 10 to 15 solvent rinses prior to beginning initial or daily calibration, especially if the instrument has set for more than several hours.

Caution: Several analytes, may be observed in the injection just following this system priming. Always run an acceptable blank prior to running any standards or samples.

8.2.5 Results should be flagged with a "P" when the values of the two columns differ by more than 40RPD.

8.3 Gas chromatographic analysis:

8.3.1 Following acceptable initial calibration and calibration verification standards, samples may be analyzed. Analysis of a mid-concentration standard every 10 samples is recommended for dirty samples. Relatively clean samples may allow for an extended sequence (not to exceed a 12-hour period or 20 samples per Method 8000B) based on analyst judgment. Each sample must be bracketed with

an acceptable initial calibration or calibration verification standards interspersed between the sample analyses. When a calibration verification standard fails to meet the ± 15% difference criteria, all samples that were injected after the last standard that last met the criteria must be re-injected. Several exceptions to this rule follow: 1) If the ending calibration verification bracket fails with percent differences > 15% above the initial calibration response and no "hits" are present in the samples, then the samples may be reported; 2) If the average of the responses for all analytes is within 15%, then the calibration has been verified.

However, the average must include all analytes in the calibration, regardless of whether they are target analytes for a specific project, and the data user must be provided with the calibration verification data or a list of those analytes that exceeded the 15% limit. The effect of using the average of the response for all analytes for calibration verification would be similar to that for the initial calibration, namely that the quantitative results for those analytes where the difference is > 15% will include a greater uncertainty.

8.3.2 Analysis of a mid-concentration standard every 12-hour shift (not to exceed 20 samples) is required.

8.3.3 Examples of GC/ECD chromatograms generated by instruments with capillary columns are presented in Attachments 2-9.

8.3.4 If the peak response is less than 2.5 times the baseline noise level, the validity of the quantitative result may be questionable. The analyst should consult with the source of the sample to determine whether further concentration of the sample is warranted by the context in which the result is to be used.

8.3.5 If the peak response exceeds the range of the highest standard, quantitatively dilute the extract and reanalyze. The calibration range is defined as the area of the level 5 peak for congeners and the sum of the 5 (level5) peak for Aroclor 1016 and 1260. For aroclors other than 1016/1260 the range is defined by the following rule. If the peak height in the pattern of a target Aroclor other than 1016 or 1260 exceeds the peak height of the highest peak in the level 5 standard of the 1016/1260 initial calibration, the sample must be diluted to where the peak height of the target analyte pattern falls below the peak height of the highest peak in the level 5 standard.

8.3.6 The identification of target analytes is based on pattern recognition for aroclors and retention time windows for congeners.

Retention time (RT) windows may either be calculated (using the 72-hour study defined in SW846 method 8000B sections 7.6.1 through 7.6.4) or set as a laboratory default. For this method SWLO uses default RT window widths instead of calculating the windows for each instrument/column. The justification for using this approach on SW846 8000 series methods can be found in section 7.6 of SW846 Method 8000B. "The following subsections describe one approach that may be used to establish retention time windows for GC and HPLC methods.

Other approaches may be employed, provided that the analyst can demonstrate that they provide performance appropriate for the intended application."

Default RT window widths are carefully selected to minimize false negatives (windows too narrow) and false positives (windows too wide). The current default windows are ± 0.03 minutes for surrogate TCX and the congeners; ±0.05 minutes for surrogate DCB and the aroclors.

Due to recent advances in "high speed gas chromatography" (and to "demonstrate performance appropriate" for this method) SWLO also uses the following criteria to help define the best default RT window.

1. The window width for each analyte peak has been calculated (using the 72-hour study defined in SW846 Method 8000B sections 7.6.1 through 7.6.4) and are narrower than the default window. This can be done on one instrument and used for all instruments that have identical columns, carrier gas, and temperature programs.
2. The default window width should be wider than ± ½ the width of the peak at half-height (in the high standard).
3. The default window width should be less than ± 2 times the width of the peak at half-height (in the high standard) and less than ± 1% of the absolute retention time.

Table 9
Retention Time Windows (± minutes)

TCX	0.03
Aroclors	0.05
Congeners	0.03
DCB	0.05

8.3.7 If compound identification or quantitation are precluded due to interference (e.g., broad, rounded peaks or ill-defined baselines are present), cleanup of the extract or replacement of the capillary column or detector may be warranted.

8.4 Quantitation of Multiple Component Analytes:

8.4.1 Residues that are mixtures of two or more components present problems in measurement. When they are found together, e.g., Aroclor 1254 and Aroclor 1260, the problem of quantitation becomes even more difficult. Suggestions are offered in the following sections for handing these mixtures.

8.4.1.1 Quantitation of mixtures of residues is difficult, but reasonable accuracy can be obtained. In many cases, the chromatogram of the residue may not exactly match that of the standard. A choice must be made as to which Aroclor or mixture of Aroclors will produce a chromatogram most similar to that of the residue.

8.4.1.2 Aroclor Quantitation--Quantitate the Aroclor residues by comparing the responses of 3 to 5 major peaks in each appropriate Aroclor standards with the peaks obtained from the Aroclor(s) in the sample extract. The amount of Aroclor is calculated using each of the major peaks, and the results of the 3 to 5 determinations are averaged. Major peaks are defined as those peaks in the Aroclor standards that are at least 25% of the height of the largest Aroclor peak and are not precluded by interference in samples containing that particular Aroclor. Later eluting Aroclor peaks are generally the most stable in the environment. Smaller peaks may be chosen for quantitation if major Aroclor and interference peaks co-elute in the samples.

8.4.1.3 When quantitating mixtures of Aroclors, choose peaks that do not coelute with other Aroclors present in the mixture. This minimizes error in quantitation.

8.4.1.4 Technical chlordane component ratios may vary from manufacturer to manufacturer and may degrade significantly in the environment. Highly degraded chlordane looks similar to degraded Aroclor 1254. Therefore, special care must be taken to determine if Aroclor 1254 or technical chlordane is present.

8.5 Aroclors or mixtures of Aroclors are reported as such. PCB congeners may be reported as specific PCB congeners or as estimated "Total PCBs".

8.6 See the standard operating procedure for the "Preventive Maintenance of Hewlett-Packard and Fisons Gas Chromatographs", SWL-OP-201, for specific GC maintenance procedures.

9.0 CALCULATIONS

9.1 Calibration Factor (CF) = $\frac{\text{Peak Area (or Height) of the Compound in the Standard}}{\text{conc. in ng/mL of compound}}$

9.2 Mean CF = $\frac{\text{Sum of CF for each level}}{\text{number of levels}}$

9.3 SD = Square root $\left[\frac{\text{Sum of (CF of each individual level - Mean CF)}^2}{\text{(number of levels - 1)}}\right]$

9.4 RSD = $\frac{\text{SD}}{\text{Mean CF}} \times 100$

Typist: LLR

9.5 See SW-846, Revision 2, January 1995, page 8000B - 20 through 22 for linear and higher order fit equations.

9.6 $$\% \text{ Difference} = \frac{\text{Mean CF - CF from the calibration verification standard}}{\text{Mean CF}} \times 100$$

9.7 $$\% \text{ Drift (used for linear and higher order fits)} = \frac{\text{Calculated conc. - Nominal conc.}}{\text{Theoretical conc.}} \times 100$$

9.8 Determine the concentration of individual compounds in the sample.

9.8.1 Sample Concentration Calculation:

9.8.1.1 Aqueous Samples

Concentration (µg/L)=[As] [V(t)][D] / [Mean CF] [V(s)]

where:
As = Area (or height) of the peak for the analyte in the sample.
V(t) = Total volume of concentrated extract (mL).
D = Dilution factor.
Mean CF = Mean calibration factor (1/ng/mL)
V(s) = Volume of aqueous sample extracted (mL).

9.8.1.2 Non-aqueous Samples

Concentration (µg/Kg)=[As] [V(t)][D] / [Mean CF] [W(s)]

where:
As = Area (or height) of the peak for the analyte in the sample.
V(t) = Total volume of concentrated extract (mL).
D = Dilution factor.
Mean CF = Mean calibration factor (1/ng/mL)
W(s) = Weight of sample extracted (g).

9.9 Report results in µg/L for water samples and µg/Kg for solid samples without correction for recovery data. All QC data obtained should be reported with the sample results.

9.10 Dry weight calculation (from data gathered during step 8.1.5.1.6):

$$\% \text{ Moisture} = \frac{\text{grams of sample - grams of dried sample}}{\text{grams of sample}} \times 100$$

10.0 QUALITY ASSURANCE/QUALITY CONTROL

10.1 Refer to Chapter One of SW-846 for specific quality control (QC) procedures. Quality control to validate sample extraction is covered in Method 3500 and in the extraction method utilized. If extract cleanup is to be performed, follow the QC in Method 3600 and in the specific cleanup method.

10.2 Calculate surrogate standard recovery on all samples, blanks, and spikes. Determine if the recovery is within limits established. If recovery is not within limits, the following are required:

10.2.1 Check to be sure there are no errors in calculations and surrogate solutions. Also, check instrument performance.

10.2.2 Recalculate the data and/or reanalyze the extract if any of the above checks reveal a problem.

10.2.3 If both method blank surrogate recoveries are outside of control limits, reanalyze the sample to rule out instrument problems. Re-extract and reanalyze the samples if none of the above are a problem.

10.3 If the spike recoveries are outside of laboratory control limits in both the LCS and LCSD, the LCS/LCSD and associated samples must be re-extracted and reanalyzed. If out-of-control analytes are out high and those analytes are not present in the samples, then no corrective action is required. Control limits will be those annually updated from in-house generated data. See "Blue Book" for SWLO's current control limits.

10.4 If spike recoveries in the MS/MSD are consistent and outside of control limits but the combined LCS/LCSD are within control limits, the poor MS/MSD recoveries may be attributed to matrix effect. If both LCS/LCSD and MS/MSD are outside of control limits the samples and associated QC must be re-extracted and reanalyzed. Control limits will be those annually updated from in-house generated data. See "Blue Book" for SWLO's current control limits.

10.5 Include a mid-concentration calibration standard (1016/1260 for Aroclors) at least every 20 or more samples (not to exceed a 12-hour period) in the analysis sequence as a calibration check. The response factors for the mid-concentration calibration should be within 15 percent of the average values for the multi-concentration calibration. When this continuing calibration is out of this acceptance window, the laboratory should stop analyses, clean the injector, replace the septum and recalibrate the system. Samples that are not preceded by acceptable standards must be re-injected to avoid errors in quantitation. Several exceptions to this rule follows: 1) <u>If the ending calibration verification bracket fails with percent differences > 15% above the initial calibration response and no "hits" are present in the samples, then the samples may be reported; 2) If the average of the responses for all analytes is within 15%, then the calibration has been verified. However, the average must include all analytes in the calibration, regardless of whether they are target analytes for a specific project, and the data user must be provided with the calibration verification data or a list of those analytes that exceeded</u>

the 15% limit. The effect of using the average of the response for all analytes for calibration verification would be similar to that for the initial calibration, namely that the quantitative results for those analytes where the difference is > 15% will include a greater uncertainty.

10.6 If any of the target analytes in the calibration verification fall outside of the retention time window established during initial calibration, the system is considered out of control. All samples analyzed after the last compliant calibration verification must be re-analyzed after instrument maintenance and re-calibration is successfully performed.

10.7 Instrument blanks must not contain any confirmed target analyte greater than ½ of the reporting limit for the associated samples. Method blanks must not contain any confirmed target analyte greater than the reporting limit for the associated samples. If contamination is noted, stop all analyses, determine the source of the contamination, remove the source, and confirm the source is gone by reanalyzing an instrument blank and/or method blank as appropriate. QC and associated samples will be re-extracted and reanalyzed if the blank contamination is present > PQL in the samples.

11.0 WASTE DISPOSAL

See Standard Operating Procedure "*Hazardous Waste Management Plan*", SWL-GA-114.

12.0 METHOD PERFORMANCE

12.1 The MDL concentrations are updated annually and are obtained using organic-free reagent water and sandy loam soil. The QA Department maintains MDL results in SWLO's "Red book". Details for determining MDLs are given in Chapter One of SW-846 and the SOP for the Definition and Determination of Detection Limits, SWL-GA-113. The MDL actually achievable in a given analysis is dependent on any matrix effects.

12.2 The accuracy and precision obtainable following this method will be determined by the sample matrix, sample preparation technique, optional cleanup techniques, and calibration procedures used.

12.3 The laboratory should maintain control charts of LCS, MS, and MSD recoveries and relative percent deviations between MS and MSD recovery for accuracy and precision data. Surrogate recovery is also plotted. The control charts are monitored monthly for LCS and quarterly for MS and are maintained on file in the laboratory. Control limits are updated on an annual basis (See SOP "Control Charts", SWL-GA-112 for more details). See "Blue Book" for SWLO's most current control limits.

12.4 Example chromatograms are shown in Attachments 2-5.

13.0 REFERENCES

U.S.EPA SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods: "Method 3500B. Organic Extraction and Sample Preparation" Revision 2, December 1996.

U.S.EPA SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods: "Method 3510C. Separatory Liquid-Liquid Extraction" Revision 3, December 1996.

U.S.EPA SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods: "Method 3520C. Continuous Liquid-Liquid Extraction" Revision 3, December 1996.

U.S.EPA SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods: "Method 3540C. Soxhlet Extraction" Revision 3, December 1996.

U.S.EPA SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods: "Method 3550B. Sonication Extraction" Revision 2, December 1996.

U.S.EPA SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods: "Method 3580A. Waste Dilution" Revision 1, July 1992.

U.S.EPA SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods: "Method 8000B. Determinative Chromatographic Separations" Revision 2, December 1996.

U.S.EPA SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods: "Method 8082. Polychlorinated Biphenyls (PCBs) by Gas Chromatography" Revision 0, December 1996.

Standard Operating Procedure, "Preventative Maintenance of Hewlett-Packard and Fisons Gas Chromatographs", SWL-OP-201.

Standard Operating Procedure, "Standards Receipt, Traceability, and Preparation", SWL-OP-202.

Standard Operating Procedure, "Laboratory Safety Plan", SWL-GA-111.

Standard Operating Procedure, "Control Charts", SWL-GA-112.

Standard Operating Procedure, "Definition and Determination of Detection Limits", SWL-GA-113.

Standard Operating Procedure, "Hazardous Waste Management Plan", SWL-GA-114.

Headquarters Air Force Center for Environmental Excellence Quality Assurance Project Plan, Version 2.0, January 1997.

U.S.EPA, Statement of Work OLM03.2, "Organic Analysis: Multi-media, Multi-concentration".

14.0 DEFINITIONS

PQL:..... Practical Quantitation Limit.

N/A:..... not applicable

Material Safety Data Sheet (MSDS): ..... Information provided by commercial vendors for the chemical products prepared by their company. The information on the MSDS includes but is not limited to composition, physical properties, hazard identification, first aid measures, emergency clean up and/or response, exposure, and storage.

SDG: ..... the Sample Delivery Group; a unit within a single case that is used to identify a group of samples for delivery. A SDG is a group of 20 or fewer field samples within a case, received over a period of up to 14 calendar days (7 calendar days for 14-day turns). Data from all samples in a SDG are due concurrently.

15.0 ATTACHMENTS

Attachment 1: ..... Sample Extraction Log

Attachment 2: ..... Chromatographic Report: Congeners

Attachment 3: ..... Chromatographic Report: AR1221

Attachment 4: ..... Chromatographic Report: AR1232

Attachment 5: ..... Chromatographic Report: AR 1242

Attachment 6: ..... Chromatographic Report: AR1248

Attachment 7: ..... Chromatographic Report: AR1254

ATTACHMENT 1
Sample Extraction Log

Typist: LLR

[redacted] | SV | [redacted] | CB | OTHER

NT EX. (520B) | | | |
P-F (510B) | | XXXXX | |
AKER | | | |
NC. (550) | | | |
XHLET | | | |

[ ] RUSH [ ] REEXTRACT [ ] TCLP

SAMPLE EXTRACTION LO

SWLO
SOUTHWEST LABORATORY OF OKLAHOMA INC.

BATCH: 991110WB

EXTRACT DATE: 1999-11-10 08:27

000646
Page: 1 of 1

SAMPLE ID (ISODE #)	CLIENT CASE	DATE RECEIVED / CLIENT SAMPLE ID	MATRIX/LEVEL	SAMPLE pH	% MOISTURE	DRY WEIGHT (g)	AMOUNT EXTRACTED (g or ml)	EXTRACTED VOLUME (ml)	GPC VOLUME (ml)	GPC ELUTE VOL (ml)	FLORISIL VOL CONC (ml)	FLORISIL FIN VOL CONC (ml)	VOL SV (P) (ml)	FIN PROCESS VOLUME (ml)	SCREEN Y/N	MS SV (P) (µL)	SURR SV (P) (µL)	BASE pH CONFIRMATION	ACID pH CONFIRMATION
L1110WB			W/L				1000	200	N\A	N\A	N\A	N\A	10.0	10.0	N		1000		
B1110WB			W/L				1000	200	N\A	N\A	N\A	N\A	10.0	10.0	N		1000		
C1110WB			W/L				1000	200	N\A	N\A	N\A	N\A	10.0	10.0	N	1000	1000		
D1110WB			W/L				1000	200	N\A	N\A	N\A	N\A	10.0	10.0	N	1000	1000		
1011.01 C801	XXXXXXXX XXXXXXXX	XXXXXXXXXXXXXXXX XXXXXXXXXXXXXXXXXXXX	W/L	6.44			1000	200	N\A	N\A	N\A	N\A	10.0	10.0	N		1000		
1011.02 C801	XXXXXXXX XXXXXXXX	XXXXXXXXXXXXXXXX XXXXXXXXXXXXXXXXXXXX	W/L	7.08			1000	200	N\A	N\A	N\A	N\A	10.0	10.0	N		1000		
1011.03 C801	XXXXXXXX XXXXXXXX	XXXXXXXXXXXXXXXX XXXXXXXXXXXXXXXXXXXX	W/L	7.09			1000	200	N\A	N\A	N\A	N\A	10.0	10.0	N		1000		
1011.04 C801	XXXXXXXX XXXXXXXX	XXXXXXXXXXXXXXXX XXXXXXXXXXXXXXXXXXXX	W/L	7.64			1000	200	N\A	N\A	N\A	N\A	10.0	10.0	N		1000		
1013.01 C801	[redacted]	11/09/1999 09:15 U9-16-014	W/L	6.80			1000	200	N\A	N\A	N\A	N\A	10.0	10.0	N		1000		
1013.02MS C801	[redacted]	11/09/1999 09:15 U9-16-014	W/L	6.80		✓	500	200	N\A	N\A	N\A	N\A	10.0	10.0	N	1000	1000		
1013.03MSD C801	[redacted]	11/09/1999 09:15 U9-16-014	W/L	6.80		✓	500	200	N\A	N\A	N\A	N\A	10.0	10.0	N	1000	1000		

INITIALS

WEIGHING:	RC	EXTRACTION:	DDC	FLORISIL:	N/A
CLEANUP:	N/A	KD:	RC		
BLOWDOWN:	CLD	TV:	N/A		
SURROG. SPIKER:	DDC	VERIFIER:	JR		
MATRIX SPIKER:	DDC	VERIFIER:	JR		
SUBMITTED BY:	CLD	REVIEWED BY:			

ID NUMBERS

FLORISIL CARTRIDGE #: N/A

GPC COLUMN #: N/A

SURROGATES ID CONC #: 5-261-4

MATRIX SPIKE SOLID/CONC #(s): 5-188-4

SOP - METHOD # : SWL-OP- 414 Rev. [redacted]

COMMENTS

✓ Insuff sample for full vol.

ATTACHMENT 2
Chromatographic Report: Congeners

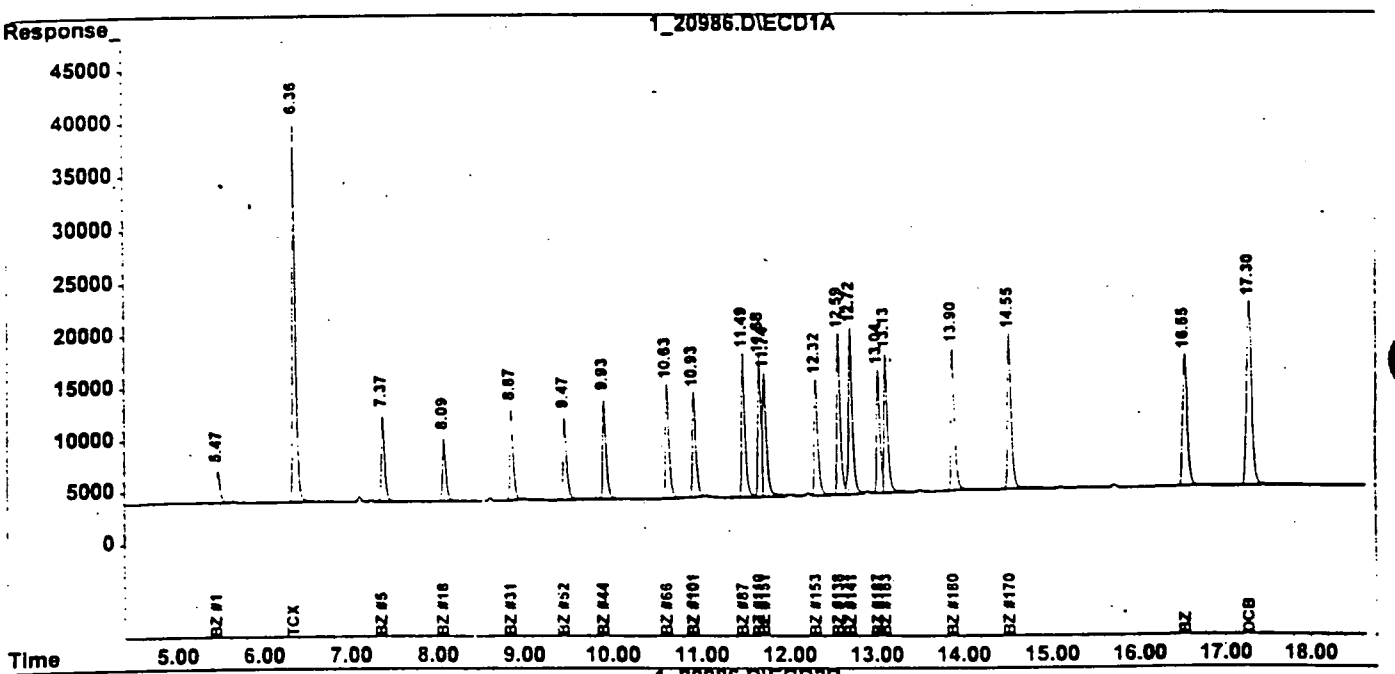
Typist: LLR

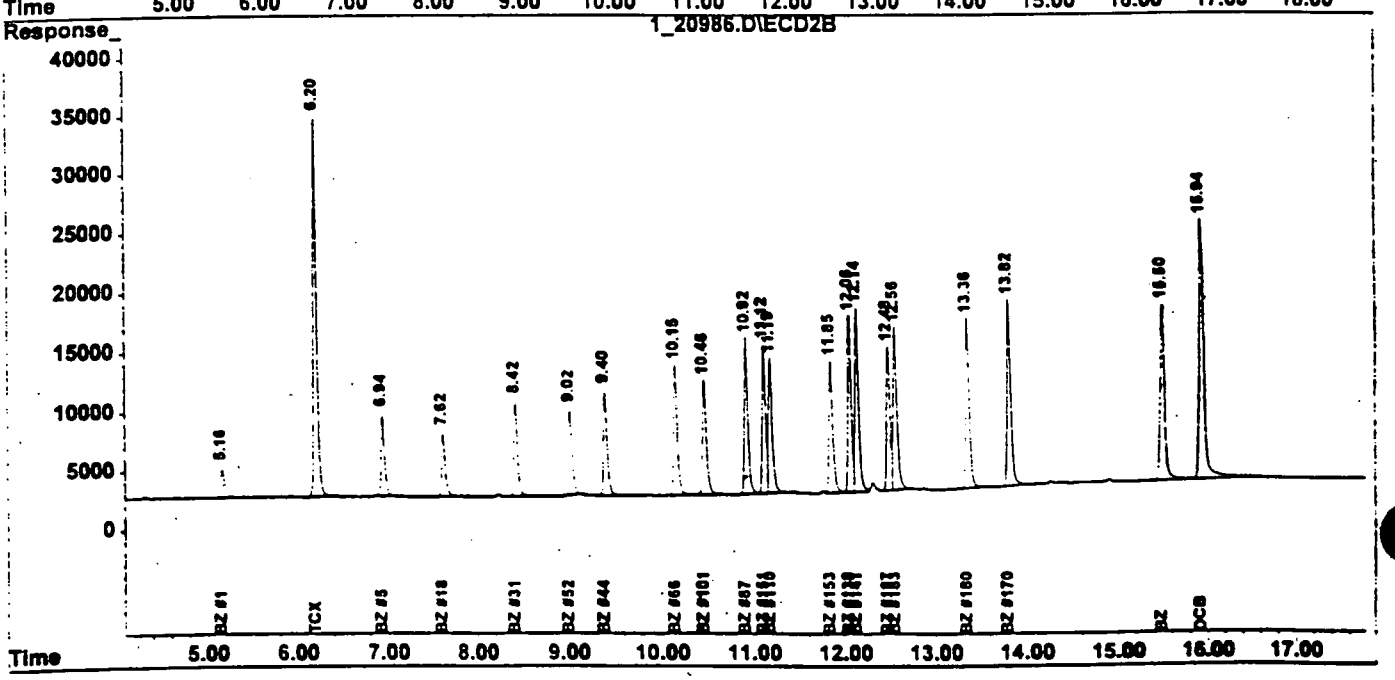
Chromatographic Report

Signal #1 : F:\HPCHEM\HP\1\DATA\10_07_99\1_20986.D\ECD1A.CH Vial: 16
Signal #2 : F:\HPCHEM\HP\1\DATA\10_07_99\1_20986.D\ECD2B.CH
Acq On : 7 Oct 1999 13:59 Operator:
Sample : BZL51A Inst : HP_01
Misc : 5-231-4 Multiplr: 1.0000
IntFile Signal #1: events.e IntFile Signal #2: events2.e
Quant Time: Oct 7 14:28 1999 Quant Results File: BZJ07.RES

Method : F:\HPCHEM\HP\1\METHODS\BZJ07.M
Title-Path : SW-846, METHOD 8082 - F:\HPCHEM\HP\1\DATA\10_07_99\
Last Update : Fri Oct 08 09:02:03 1999
Response via : Multiple Level Calibration

Volume Inj. : 1uL
Signal #1 Phase : DB-1701 Signal #2 Phase: DB-17
Signal #1 Info : .32 Signal #2 Info : .32
Signal #1 Inst : HP_01A Signal #2 Inst : HP_01B





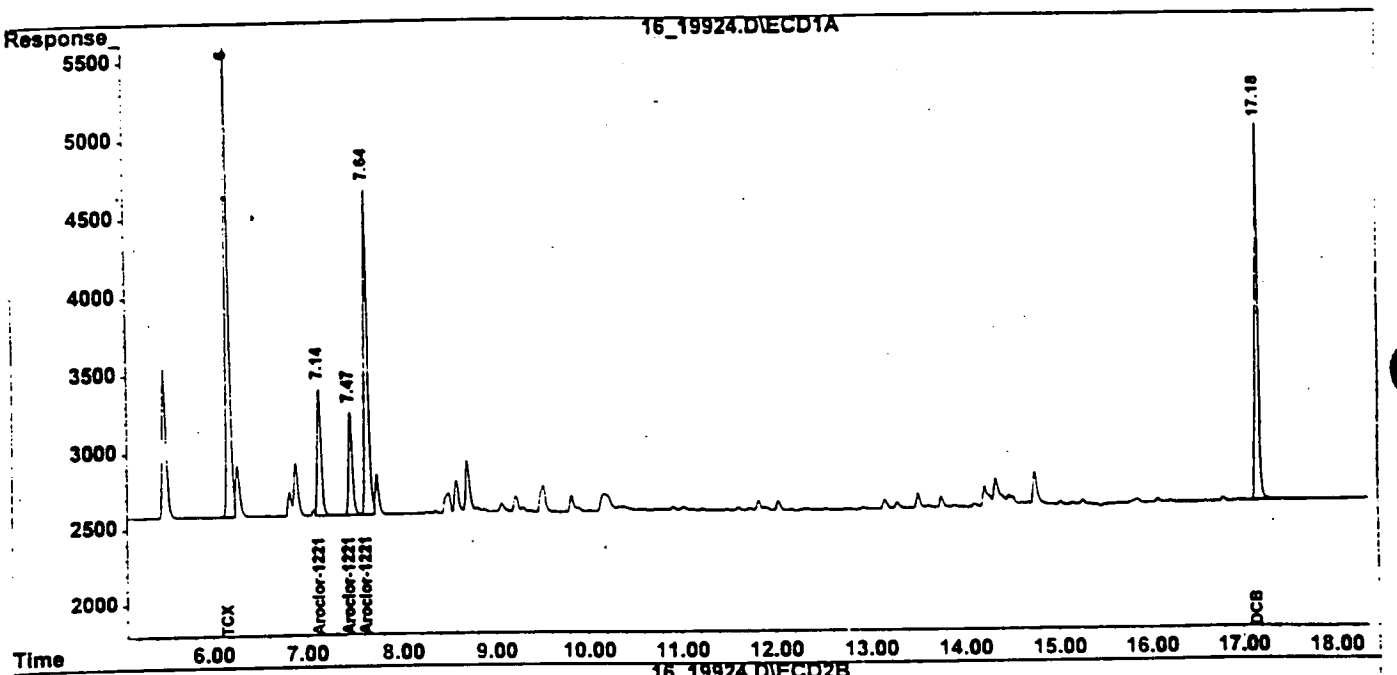
ATTACHMENT 3
Chromatographic Report: AR1221

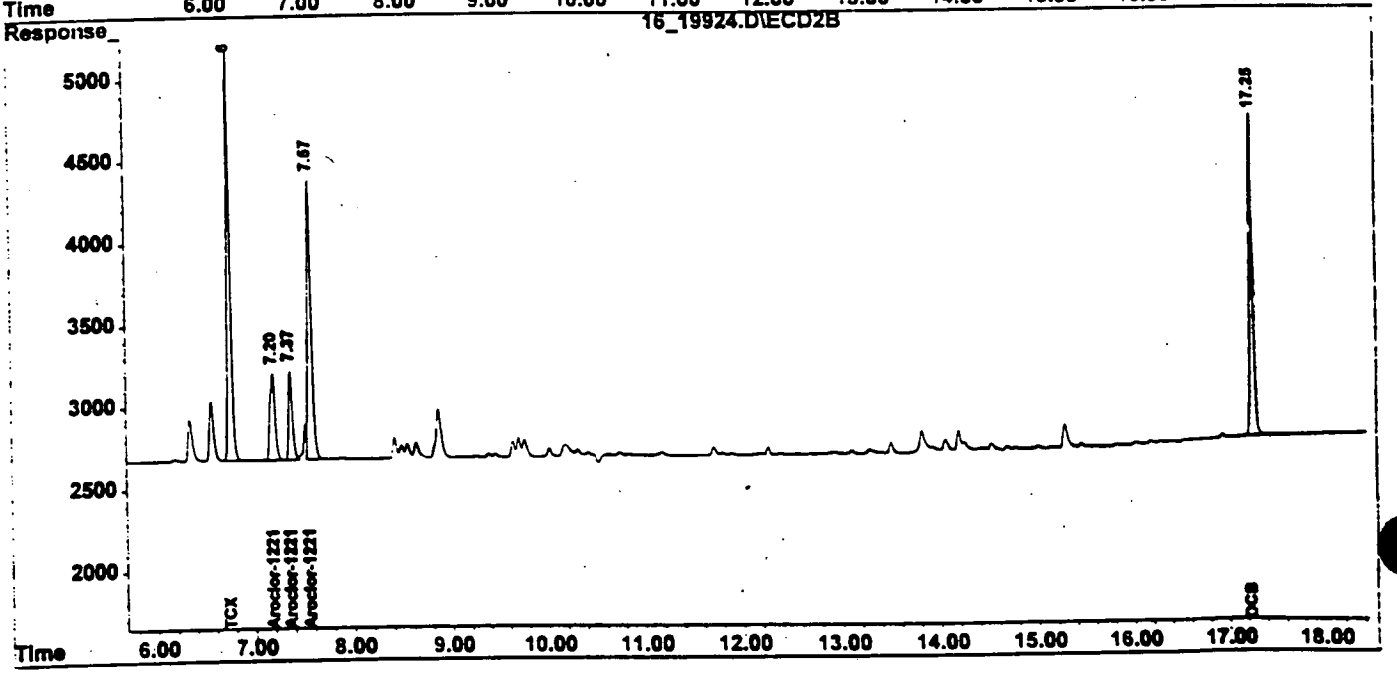
Chromatographic Report

Signal #1 : F:\HPCHEM\HP\16\DATA\10_26_99\16_19924.D\ECD1A.CH Vial: 7
Signal #2 : F:\HPCHEM\HP\16\DATA\10_26_99\16_19924.D\ECD2B.CH
Acq On : 26 Oct 1999 16:26 Operator: NITA
Sample : AR1221L1160 Inst : HP_16
Misc : 5-62-4 Multiplr: 1.0000
IntFile Signal #1: events.e IntFile Signal #2: events2.e
Quant Time: Oct 27 7:46 1999 Quant Results File: SPPCBJ26.RES

Method : F:\HPCHEM\HP\16\METHODS\SPPCBJ26.M
Title-Path : PCB/TOXAPHENE - F:\HPCHEM\HP\16\DATA\10_21_99\
Last Update : Thu Oct 28 14:57:34 1999
Response via : Single Level Calibration

Volume Inj. : 0.5uL
Signal #1 Phase : DB-17MS Signal #2 Phase: DB-XLB
Signal #1 Info : .25 Signal #2 Info : .25
Signal #1 Inst : HP_16A Signal #2 Inst : HP_16B





ATTACHMENT 4
Chromatographic Report: AR1232

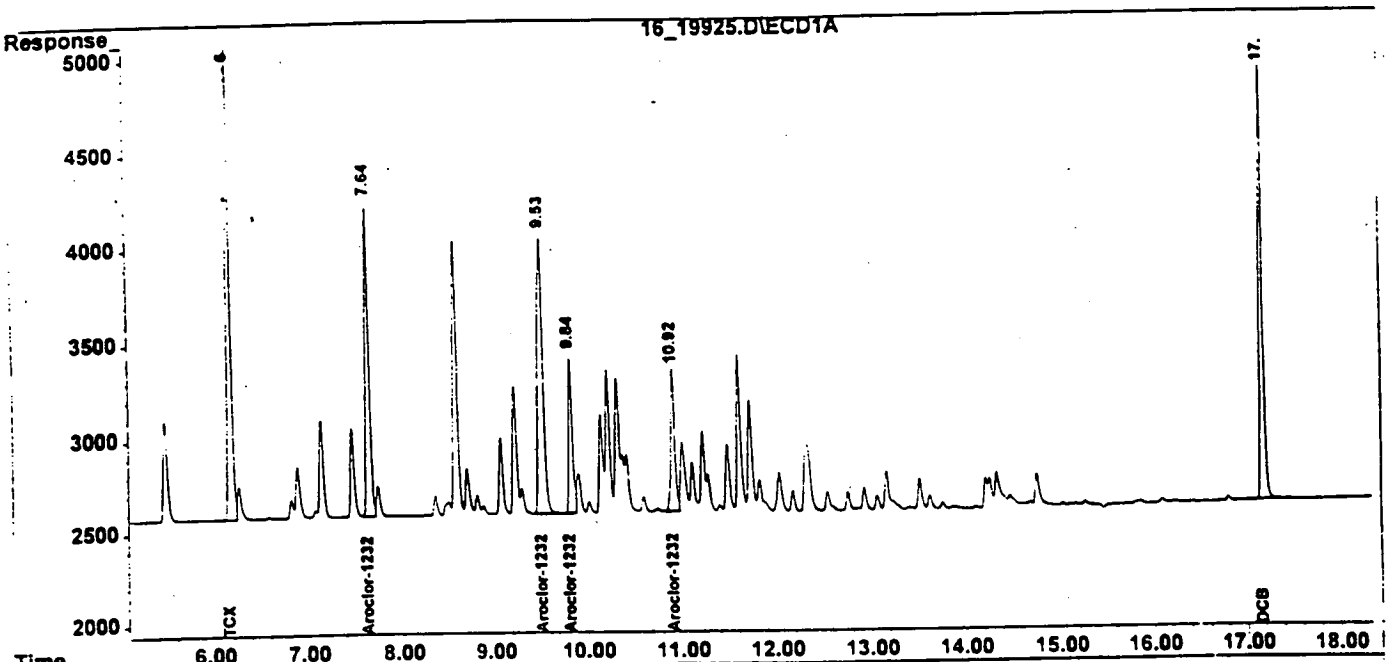
Chromatographic Report

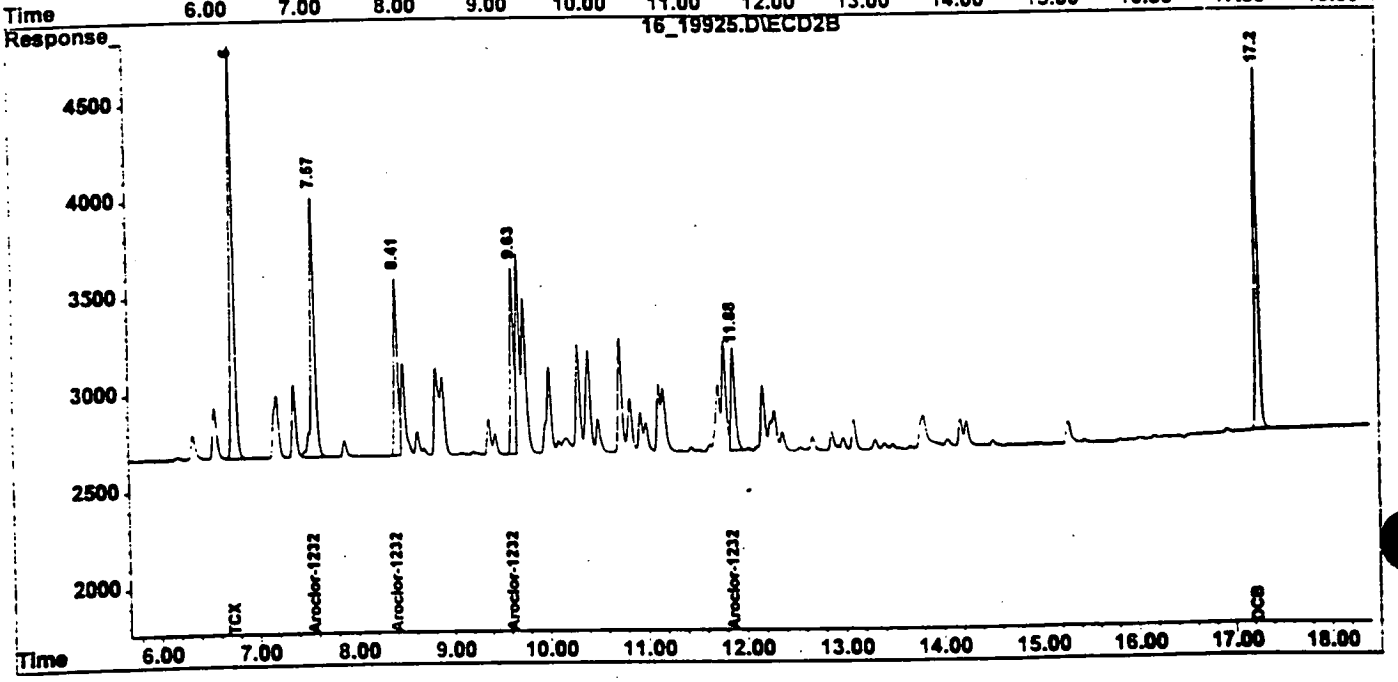
Signal #1 : F:\HPCHEM\HP\16\DATA\10_26_99\16_19925.D\ECD1A.CH Vial: 8
Signal #2 : F:\HPCHEM\HP\16\DATA\10_26_99\16_19925.D\ECD2B.CH
Acq On : 26 Oct 1999 16:49 Operator: NITA
Sample : AR1232L1160 Inst : HP_16
Misc : 5-67-4 Multiplr: 1.0000
IntFile Signal #1: events.e IntFile Signal #2: events2.e
Quant Time: Oct 27 7:46 1999 Quant Results File: SPPCBJ26.RES

Method : F:\HPCHEM\HP\16\METHODS\SPPCBJ26.M
Title-Path : PCB/TOXAPHENE - F:\HPCHEM\HP\16\DATA\10_26_99\
Last Update : Thu Oct 28 14:57:34 1999
Response via : Single Level Calibration

Volume Inj. : 0.5uL
Signal #1 Phase : DB-17MS
Signal #1 Info : .25
Signal #1 Inst : HP_16A

Signal #2 Phase: DB-XLB
Signal #2 Info : .25
Signal #2 Inst : HP_16B





ATTACHMENT 5
Chromatographic Report: AR1242

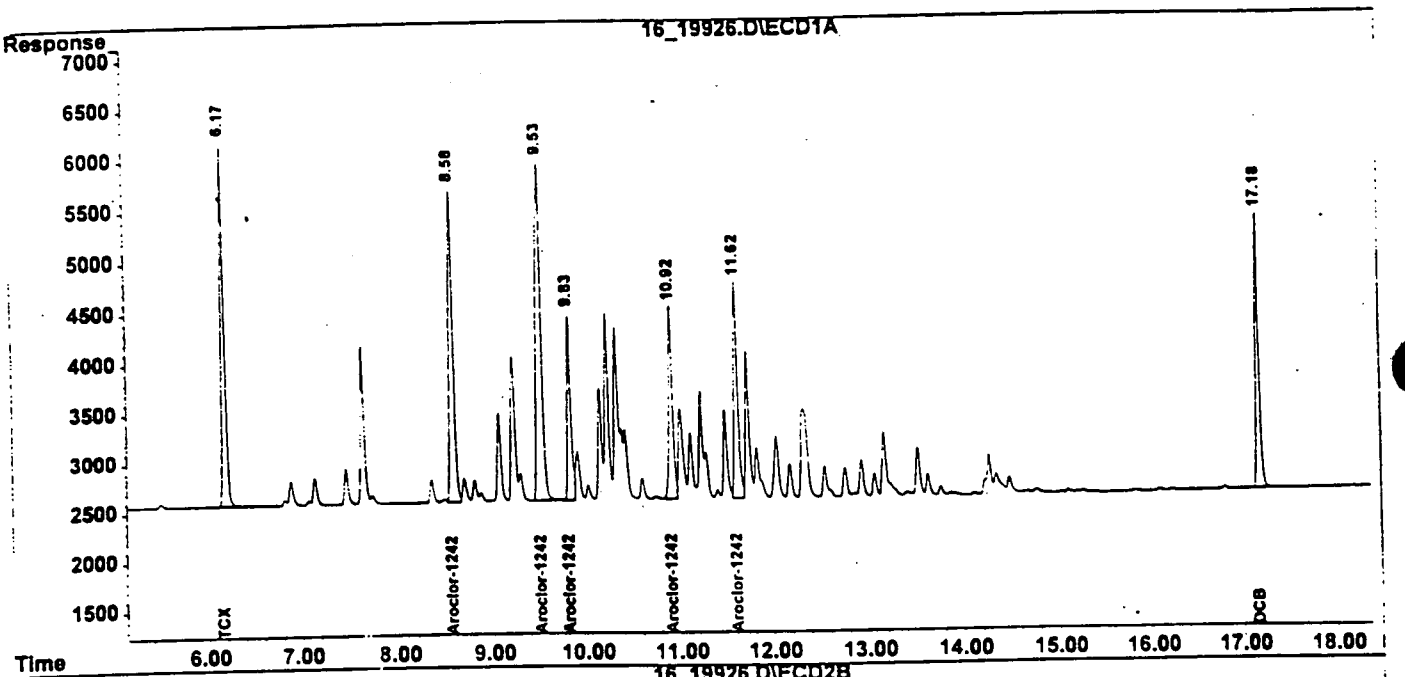
Chromatographic Report

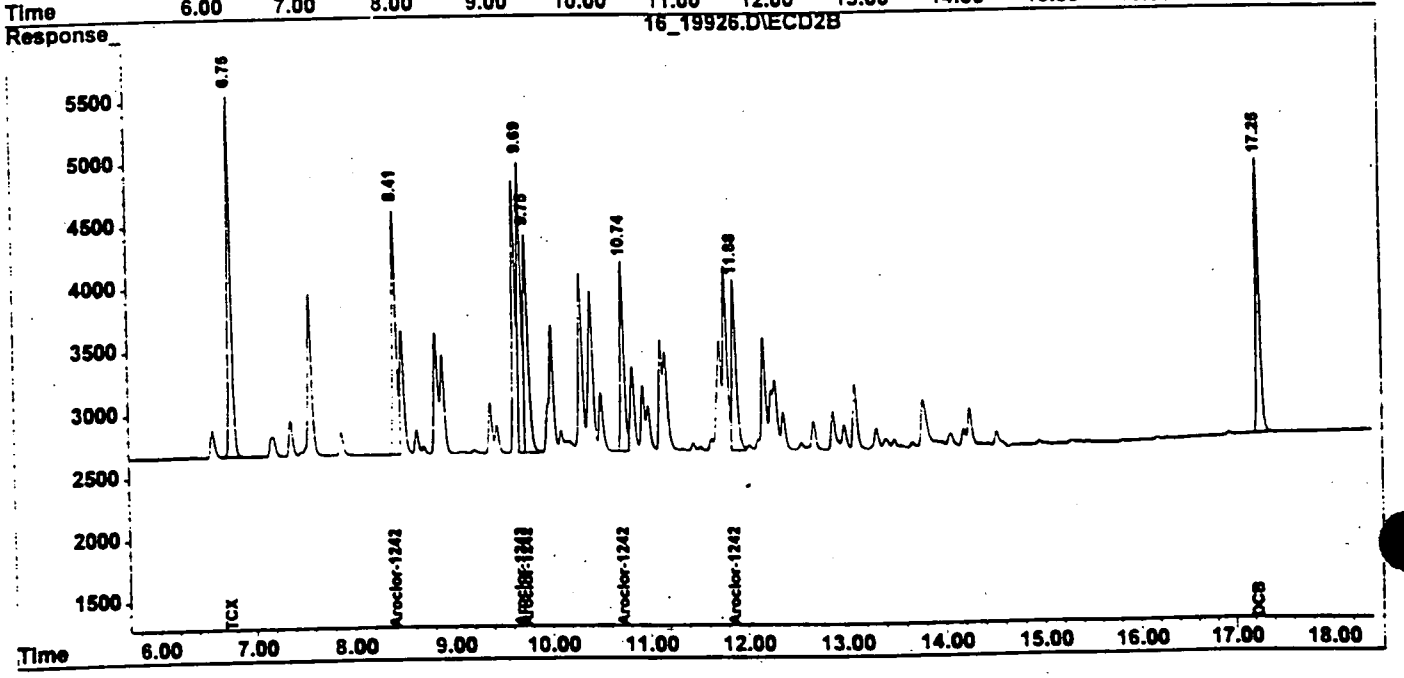
Signal #1 : F:\HPCHEM\HP\16\DATA\10_26_99\16_19926.D\ECD1A.CH Vial: 9
Signal #2 : F:\HPCHEM\HP\16\DATA\10_26_99\16_19926.D\ECD2B.CH
Acq On : 26 Oct 1999 17:12 Operator: NITA
Sample : AR1242L1160 Inst : HP_16
Misc : 5-265-3 Multiplr: 1.0000
IntFile Signal #1: events.e IntFile Signal #2: events2.e
Quant Time: Oct 27 7:47 1999 Quant Results File: SPPCBJ26.RES

Method : F:\HPCHEM\HP\16\METHODS\SPPCBJ26.M
Title-Path : PCB/TOXAPHENE - F:\HPCHEM\HP\16\DATA\10_26_99\
Last Update : Thu Oct 28 14:57:34 1999
Response via : Single Level Calibration

Volume Inj. : 0.5uL
Signal #1 Phase : DB-17MS
Signal #1 Info : .25
Signal #1 Inst : HP_16A

Signal #2 Phase: DB-XLB
Signal #2 Info : .25
Signal #2 Inst : HP_16B





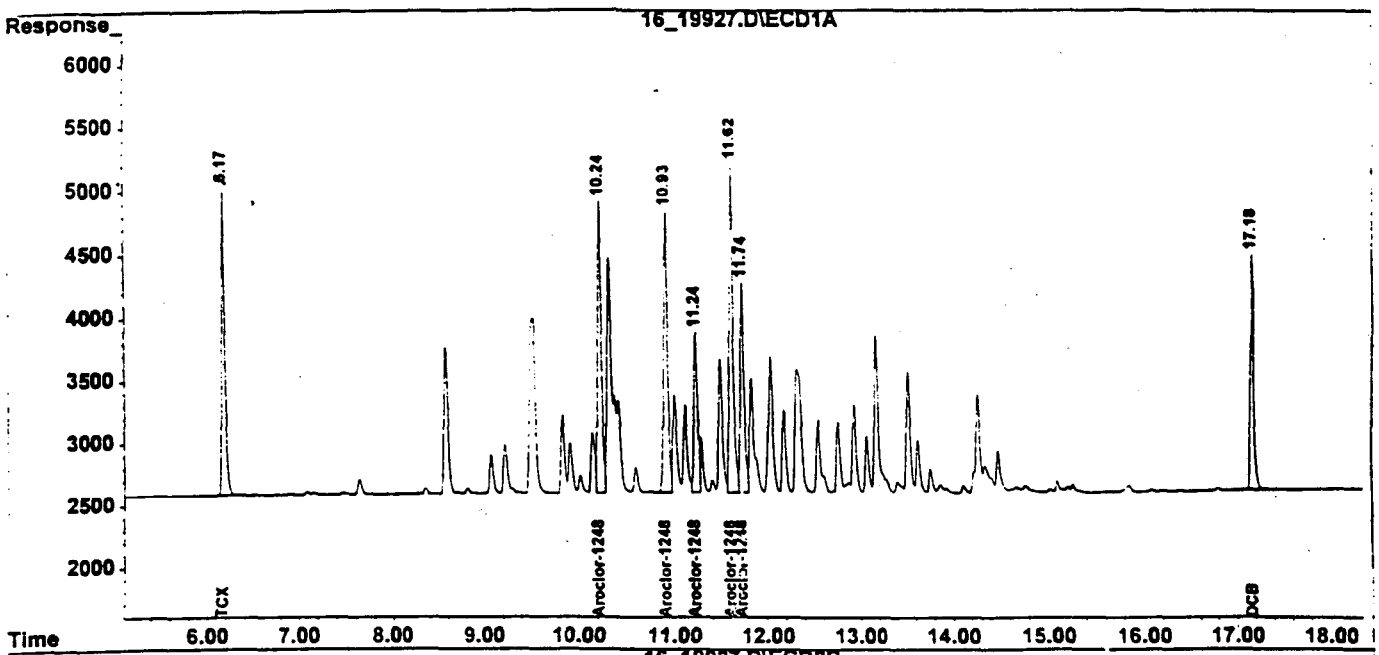
ATTACHMENT 6
Chromatographic Report: AR1248

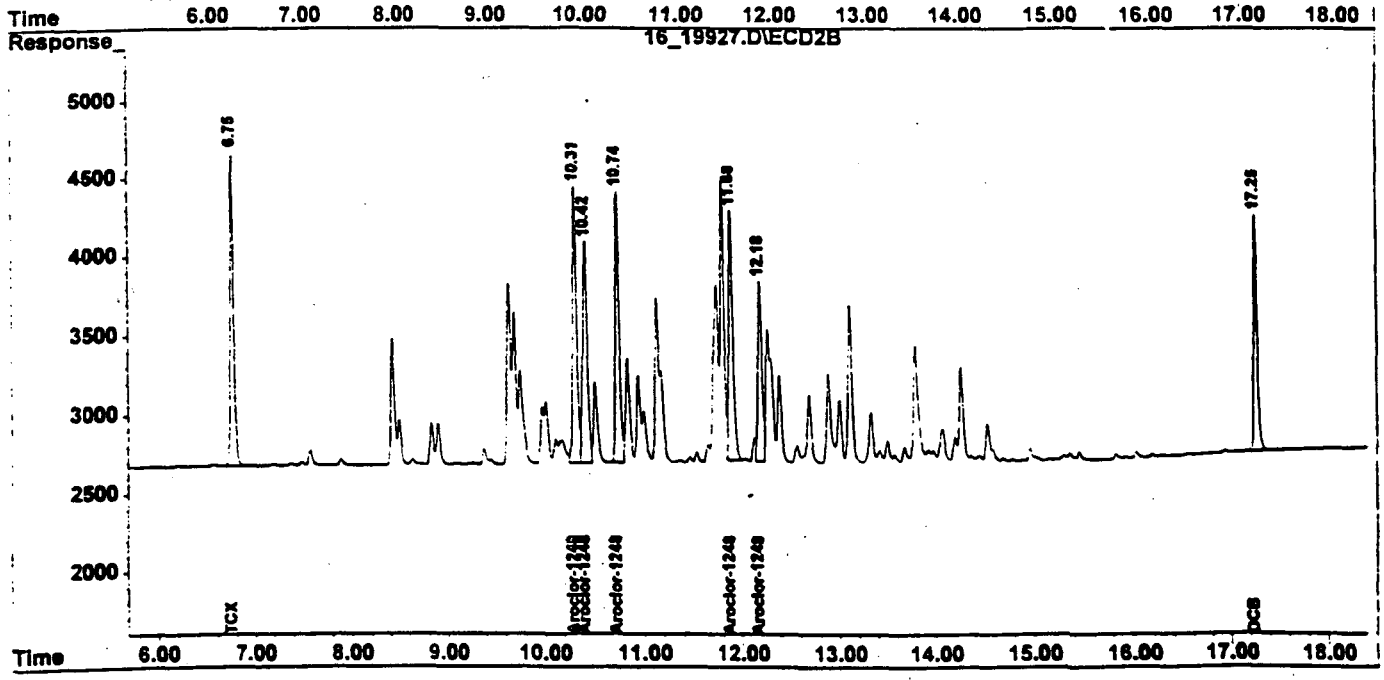
Chromatographic Report

Signal #1 : F:\HPCHEM\HP\16\DATA\10_26_99\16_19927.D\ECD1A.CH Vial: 10
Signal #2 : F:\HPCHEM\HP\16\DATA\10_26_99\16_19927.D\ECD2B.CH
Acq On : 26 Oct 1999 17:35 Operator: NITA
Sample : AR1248L1160 Inst : HP_16
Misc : 5-72-4 Multiplr: 1.0000
IntFile Signal #1: events.e IntFile Signal #2: events2.e
Quant Time: Oct 27 7:47 1999 Quant Results File: SPPCBJ26.RES

Method : F:\HPCHEM\HP\16\METHODS\SPPCBJ26.M
Title-Path : PCB/TOXAPHENE - F:\HPCHEM\HP\16\DATA\10_26_99\
Last Update : Thu Oct 28 14:57:34 1999
Response via : Single Level Calibration

Volume Inj. : 0.5uL
Signal #1 Phase : DB-17MS Signal #2 Phase: DB-XLB
Signal #1 Info : .25 Signal #2 Info : .25
Signal #1 Inst : HP_16A Signal #2 Inst : HP_16B





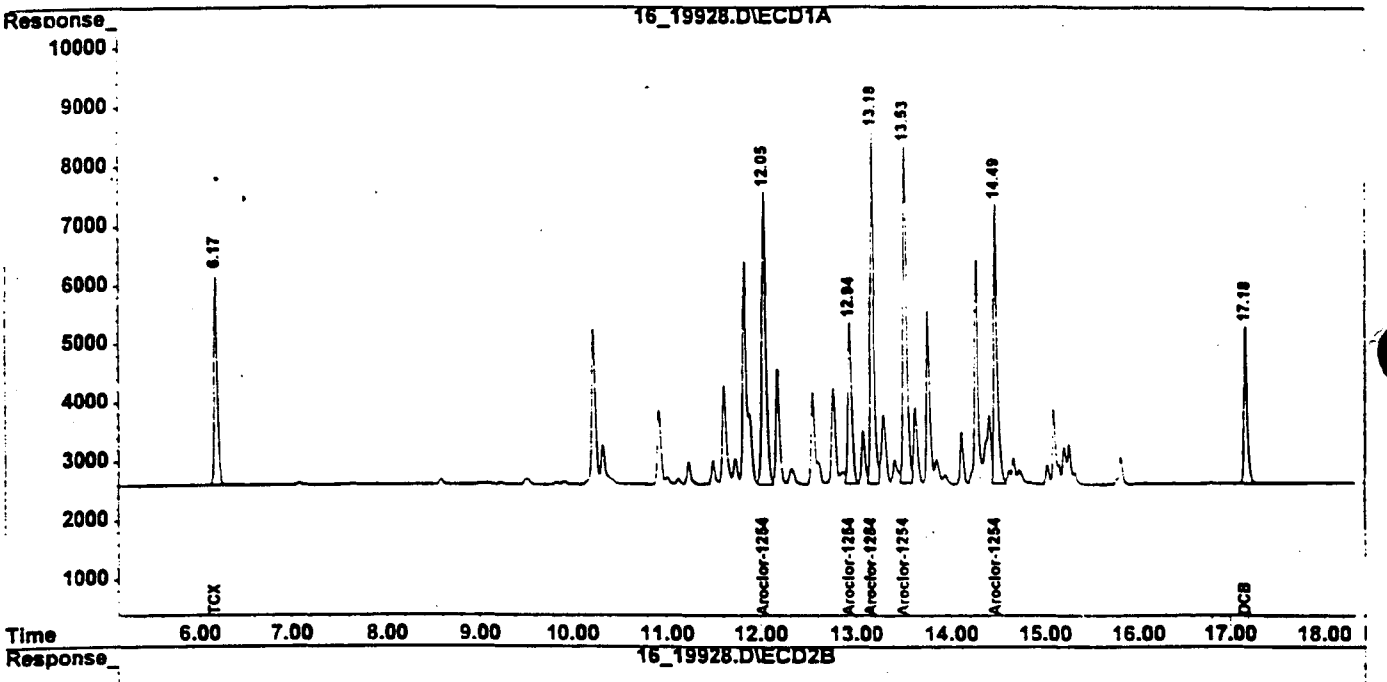
ATTACHMENT 7
Chromatographic Report: AR1254

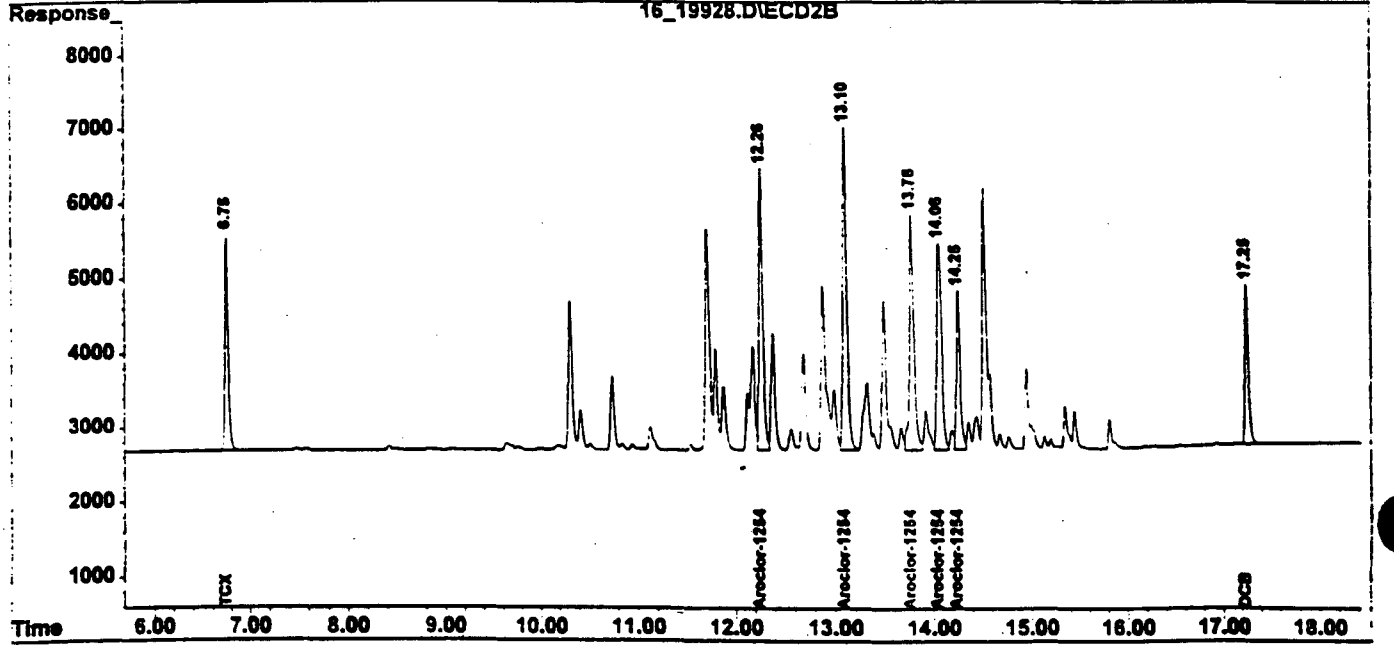
Chromatographic Report

Signal #1 : F:\HPCHEM\HP\16\DATA\10_26_99\16_19928.D\ECD1A.CH Vial: 11
Signal #2 : F:\HPCHEM\HP\16\DATA\10_26_99\16_19928.D\ECD2B.CH
Acq On : 26 Oct 1999 17:58 Operator: NITA
Sample : AR1254L1160 Inst : HP_16
Misc : 5-275-3 Multiplr: 1.0000
IntFile Signal #1: events.e IntFile Signal #2: events2.e
Quant Time: Oct 27 7:48 1999 Quant Results File: SPPCBJ26.RES

Method : F:\HPCHEM\HP\16\METHODS\SPPCBJ26.M
Title-Path : PCB/TOXAPHENE - F:\HPCHEM\HP\16\DATA\10_26_99\
Last Update : Thu Oct 28 14:57:34 1999
Response via : Single Level Calibration

Volume Inj. : 0.5uL
Signal #1 Phase : DB-17MS Signal #2 Phase: DB-XLB
Signal #1 Info : .25 Signal #2 Info : .25
Signal #1 Inst : HP_16A Signal #2 Inst : HP_16B





SOUTHWEST LABORATORY OF OKLAHOMA, INC. *and*
AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.

Standard Operating Procedure

Organic Data Package Assembly

Document No.: SWL-OS-301

Rev. No. / Date: Rev. 4.0 — 10/01/99

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APPROVALS	DATES
Harry M. Boro Procedure Prepared By	10-1-99 Date
Harry M. Boro Program Manager or Section Supervisor	10-1-99 Date
Chuck Young Laboratory Safety Officer	10/1/99 Date
Chuck Young Laboratory QA/QC Officer	10/1/99 Date

(Effective Date is 5 calendar days after the last signature above - QA/QC Officer)

Document Status

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[✓] Official Copy OC No.: 2382 Issued to: Tom Johnston Date: 05/05/00 QA: [illegible]

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ORGANIC DATA PACKAGE ASSEMBLY

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1.0 **SCOPE AND APPLICATION.** The purpose of this Standard Operating Procedure is to provide guidelines for the assembly of GC and GC/MS data per the USEPA "Contract Laboratory Program" CLP protocol.

2.0 METHOD SUMMARY

2.1 Once sample receiving has logged in all samples and generated LIMS Sample Worksheets for the case, the paperwork is sent to the data assembly room. A Case Packet is made, which consists of any forms or data pertaining to the case, such as the Chain of Custody, Traffic Report, Sample Tags, worksheets, etc. A Case Packet Form is attached to the packet and it is filed in the "In House" files.

2.2 The GC/MS operator sends the raw data for a particular case along with all the QC requirements to the data reviewer, and after review the data is sent for data assembly.

2.3 All data for each sample should be kept together. All extra (non-usable) data should be together and noted as such. The data is then given to the Data Assembly Department.

2.4 Summary of Data Assembly Steps:

2.4.1 **Operators.** As data comes off instruments, operators check the data to see if the data meets the QC requirements. Operators check Target automated Forms II, III, V, and VIII. If the QC requirements are met, the data then goes for data review. If the QC requirements are not met, then the operator takes the appropriate action.

2.4.2 **Data Reviewers.** The data then goes to the Data Reviewers who check that all requirements requested are met. If not, the data is given back to the operator for appropriate action. When all data is correct and reviewed, it then goes to the data department.

2.4.3 **Data Assembly Clerk.** Using the Envision Program, the data clerks generate all the forms and the diskettes. The data is arranged as per the delivery requirements. All the forms, header information, and spectra, are checked, the pages numbered and the copies made. Hard copies of all manual integration are accounted for and included with the data.

2.4.4 **Section Supervisors.** The data is reviewed again, the SDG narrative is written, signed, and the data package is given to the Data Clerks for shipping.

2.4.5 **Data Assembly Clerk:** The pages of packages are paginated and the appropriate number of 2-sided copies are made. The copies are bound and Federal Expressed to the locations designated by IFB instructions. A copy is retained in-house. The full CSF (original) is sent to the Region.

2.4.6 **Error Correction.** It is the responsibility of data handlers to correct errors in an acceptable manner: line out the error, initial and date. WHITE OUT AND OBLITERATIONS ARE NOT ALLOWED!

3.0 HEALTH AND SAFETY. N/A

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE. N/A

5.0 INTERFERENCE AND POTENTIAL PROBLEMS. N/A

6.0 EQUIPMENT/APPARATUS

6.1 Target 3.0 or Equivalent

6.2 Envision 3.0 or Equivalent

6.3 Ingres 6.3/02P

6.4 Unix A·09·01

6.5 IBM Personal Wheelwriter

6.6 Manual Paginating Machine

6.7 PC Microsoft Word

6.8 Kodak 2110 Copier

6.9 LIMS

7.0 REAGENTS. N/A

8.0 PROCEDURE

8.1 Sample Traffic Reports

8.1.1 Original Sample Traffic Report page marked "Lab Copy for Return to SMO" with lab receipt information and signed in original Contractor signature, for each sample in the Sample Delivery Group.

8.1.2 Traffic Reports (TRs) shall be submitted in Sample Delivery Group (SDG) sets (i.e.; TRs for all samples in an SDG shall be clipped together), with an SDG Cover Sheet attached.

8.1.3 The SDG Cover Sheet shall contain the following items:

8.1.3.1 Lab name

8.1.3.2 Contract number

8.1.3.3 Sample Analysis Price - full sample price from contract.

8.1.3.4 Case Number

8.1.3.5 List of EPA sample numbers of all samples in the SDG, identifying the first and last samples received, and their dates of receipt (LRDs). NOTE: When more than one sample is received in the first or last SDG shipment, the "first" sample received would be the lowest sample number (considering both alpha and numeric designations); the "last" sample received would be the highest sample number (considering both alpha and numeric designations).

8.1.4 In addition, each Traffic Report must be clearly marked with the SDG Number, the sample number of the first sample in the SDG (as described in the following paragraph). This information should be entered below the Lab Receipt Date on the TR. In addition, the TR for the last sample received in the SDG must be clearly marked "SDG - FINAL SAMPLE."

8.1.5 The EPA sample number of the first sample received in the SDG is the SDG number. When several samples are received together in the first SDG shipment, the SDG number shall be the lowest sample number (considering both alpha and numeric designations) in the first group of samples received under the SDG. (The SDG number is also reported on all data reporting forms.)

8.1.6 If samples are received at the laboratory with multi-sample Traffic Reports, all the samples on one multi-sample TR may not be necessarily in the same SDG. In this instance, the laboratory must make the appropriate number of photocopies of the TR, and submit one copy with each SDG cover sheet, with each page copied clearly labeled with where the original is located.

8.2 Sample Data Summary Package

8.2.1 As specified in the Delivery Schedule, one sample Data Summary Package shall be delivered to CLASS concurrently with delivery of other required sample data. The Sample Data Summary Package consists of copies of specified items from the Sample Data Package. These items are listed below and described under part 8.2.3, Sample Data Summary Package.

8.2.2 The Sample Data Summary Package shall be ordered as follows and shall be submitted separately (i.e.; separated by rubber bands, clips or other means) directly preceding the Sample Data Package. Sample data forms shall be arranged in increasing EPA sample number order, considering both letters and numbers. BE400 is a lower sample number than BF200, as E precedes F in the alphabet.

8.2.3 The Sample Data Summary Package shall contain data for samples in one Sample Delivery Group of the Case, as follows:

8.2.3.1 SDG Narrative.

8.2.3.2 By fraction (VOA, SVOA, and PEST/PCBs) and by phase unit within each fraction — tabulated target compound results (Form I) and

tentatively identified compounds (Form I, TIC) (VOA and SVOA only).

8.2.3.3 By fraction (VOA, SVOA, and PEST/PCBs) — System Monitoring Compound results (Form II).

8.2.3.4 By fraction (VOA, SVOA, and PEST/PCBs) — matrix spike results (Form III)

8.2.3.5 By fraction (VOA, SVOA, and PEST/PCBs) — blank data (Form IV) and tabulated results (Form I) including tentatively identified compounds (Form I, TIC) (VOA and SVOA only).

8.2.3.6 Form VIIIs Internal Standard Recovery

8.3 Sample Data Package.

8.3.1 The sample data package is divided into the five major units described below. The last three units are each specific to an analytical fraction (volatiles, extractables, arochlors). If the analysis of a fraction is not required, then that fraction-specific unit is not required as a deliverable.

8.3.2 The Sample Data Package shall include data for analyses of all samples in one Sample Delivery Group, including field samples, reanalyses, blanks, and control matrix spikes.

8.3.2.1 SDG Narrative

8.3.2.1.1 The document shall be clearly labeled "SDG Narrative" and shall contain: laboratory name; Case Number; sample numbers in the Sample Delivery Group (SDG), differentiating between initial analyses and re-analyses; SDG number; contract number; and detailed documentation of any quality control, sample, shipment and/or analytical problems encountered in processing the samples reported in the data package.

8.3.2.1.2 Whenever data from sample re-analyses are submitted, the Contractor shall state in the SDG Narrative for each re-analysis, whether it considers the re-analysis to be billable, and if so, why.

8.3.2.1.3 The Contractor must also include documentation of any internal quality control processes used, a summary of corrective actions taken, and the resolution.

8.3.2.1.4 The SDG Narrative shall contain the following statement, verbatim: "I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the

conditions detailed above. Release of the data contained in this hardcopy data package and in the computer–readable data submitted on diskette has been authorized by the laboratory manager or his designee, as verified by the following signature." This statement shall be directly followed by signature of the laboratory manager or his designee with a typed line below it containing the signer's name and title, and the date of signature.

8.3.2.1.5 The SDG Narrative itself must be signed in original signature by the Laboratory manager or his designee and dated.

8.3.2.2 Traffic Reports

8.3.2.2.1 A copy of the Sample Traffic Reports submitted in Item 8.1.1 for all of the samples in the SDG. The Traffic Reports shall be arranged in increasing EPA sample number order, considering both letter and numbering in ordering samples.

8.3.2.2.2 If samples are received at the laboratory with multi-sample Traffic Reports, one copy of all such TR's for the samples in the data package must be submitted in place of individual TR's. If multi-sample TR's are used, the order of TR's should be based on the first of the EPA sample numbers that appear on each multi-sample TR.

8.3.2.3 Volatiles Data

8.3.2.3.1 QC Summary

8.3.2.3.1.1 System Monitoring Compound Recovery Summary (Form II VOA)

8.3.2.3.1.2 Control Matrix Spike Summary (Form III VOA)

8.3.2.3.1.3 Method Blank Summary (Form IV VOA) (If more than a single form is necessary, forms must be arranged in chronological order by date of analysis of the blank.)

8.3.2.3.1.4 GC/MS Tuning and Mass Calibration (Form V VOA) BFB in chronological order; by instrument.

8.3.2.3.1.5 Internal Standard Area and RT Summary (Form VIII VOA) in chronological order, by instrument.

8.3.2.3.2 Sample Data

8.3.2.3.2.1 Sample data shall be arranged in packets with the Organic Analysis Data Sheet (Form I-VOA, including Form I-VOA TIC), followed by the raw data for volatile samples. These sample packets should then be placed in increasing EPA sample number order, considering both letters and numbers in ordering samples. Phase units within a sample should be placed in order by phase unit suffix (i.e. AB123-11, AB123-12, etc.)

8.3.2.3.2.2 TCL Results — Volatile Analysis Data Sheet (Form I-VOA).

Tabulated results (identification and quantitation) of the specified target compounds (Exhibit C) of SOW OLM03.2. The validation and release of these results is authorized by a specific, signed statement in the SDG Narrative (reference 8.3.2.1). In the event that the Laboratory Manager cannot validate all data reported for each sample, the Laboratory Manager shall provide a detailed description of the problems associated with the sample in the SDG Narrative.

On Form I, the appropriate concentration units shall be either µg/L or µg/Kg. No other units are acceptable. NOTE: Report analytical results to one significant figure if the value is less than 10; to two significant figures above 10.

8.3.2.3.2.3 Tentatively Identified Compounds (Form I-VOA TIC).

This form must be included even if no compounds are found. If so, indicate this on the form by entering "0" in the field for "Number found."

Form I VOA-TIC is the tabulated list of the highest probable match for up to 30 of the nonsurrogate organic compounds not listed in Exhibit C (TCL), including the CAS (Chemical Abstracts Registry) number,

tentative identification and estimated concentration. For estimating the concentration, assume a response factor of 1 and estimate the concentration by comparison of the compound peak height or total area count to the peak height or total area count of the nearest internal standard free of interferences on the reconstructed ion chromatogram. NOTE: The laboratory must be consistent (i.e.; use peak height of all comparisons or use total area count for all comparisons).

8.3.2.3.2.4 Reconstructed total ion chromatograms (RIC) for each sample or sample extract.

RICs must be normalized to the largest nonisolvent component, and must contain the following header information:

- EPA sample number
- Date and time of analysis
- GC/MS instrument ID
- Lab file ID

Internal standard and surrogate spiking compounds are to be labeled with the names of compounds, either directly out from the peak, or on a print-out of retention times if retention times are printed over the peak. If automated data system procedures are used for preliminary identification and/or quantification of the Target Compound List (TCL) compounds, the complete data system report must be included in all sample data packages, in addition to the reconstructed ion chromatogram. The complete data system report shall include all of the information listed below. For laboratories which do not use the automated data system procedures, a laboratory "raw data sheet," containing the following information, must be included in the sample data package in addition to the chromatogram.

- EPA sample number
- Date and time of analysis
- RT of identified TCL compounds
- Ion used for quantitation with measured area
- Copy of area table form data system
- GC/MS instrument ID
- Lab file ID
- Analyst ID

8.3.2.3.2.5 For each sample, by each compound identified:

- Copies of raw spectra and copies of background-subtracted mass spectra of target compounds listed in Exhibit C of SOW OLM03.2 (TCL) that are identified in the sample and corresponding background-subtracted TCL standard mass spectra. Spectra must be labeled with EPA sample number, lab file ID, data and time of analysis, and GC/MS instrument ID; compound names must be clearly marked on all spectra.
- Copies of mass spectra of non-surrogate organic compounds not listed in Exhibit C of SOW OLM03.2, TCL (Tentatively Identified Compounds) with associated best-match spectra (three best matches), labeled as in 8.3.2.3.2.5 above.

8.3.2.3.3 Standards Data

8.3.2.3.3.1 Initial Calibration Data (Form VI VOA) - in order by instrument, if more than one instrument used.

- VOA standard(s) reconstructed ion chromatograms and quantitation reports (or legible facsimile) for the initial (five

point) calibration, labeled as in 8.3.2.3.2.4 above. Spectra are not required.

- All initial calibration data must be included, regardless of when it was performed and for which case. When more than one initial calibration is performed, the data must be put in chronological order, by instrument.

8.3.2.3.3.2 Continuing Calibration (Form VII VOA) - in order by instrument, if more than one instrument used.

- VOA standard(s) reconstructed ion chromatograms and quantitation reports (or legible facsimile) for all continuing (12 hour) calibrations, labeled as in 8.3.2.3.2.4 above. Spectra are not required.

- When more than one continuing calibration is performed, forms, must be in chronological order, by instrument.

8.3.2.3.4 Raw QC Data

8.3.2.3.4.1 BFB (for each 12-hour period, for each GC/MS system utilized)

- Bar graph spectrum, labeled as in 8.3.2.3.2.4 above.

- Mass listing, labeled as in 8.3.2.3.2.4 above.

- Reconstructed Total Ion Chromatogram — labeled as in 8.3.2.3.2.4 above.

8.3.2.3.4.2 Blank Data — arranged by type (i.e., method, storage, instrument), in chronological order by instrument. NOTE: This order is different from that used for samples.

- Tabulated results (Form I)

- Tentatively Identified Compounds (Form I VOA-TIC even if none found).

- Reconstructed ion chromatogram(s) and quantitation report(s) or legible facsimile (GC/MS), labeled as in 8.3.2.3.2.4 above.
- TCL spectra with lab generated standard, labeled as in 8.3.2.3.2.5 above. Data systems which are incapable of dual display shall provide spectra in order:
  - Raw TCL compound spectra
  - Enhanced or background subtracted spectra
  - Laboratory generated TCL standard spectra
- GC/MS library search spectra for Tentatively Identified Compounds (TIC), labeled as in 8.3.2.3.2.5 above.
- Quantitation/Calculation of Tentatively Identified Compound(s) (TIC) concentrations

8.3.2.3.4.3 Matrix Spike and Duplicate Data

- Tabulated results (Form I VOA) TCL compounds. Form I VOA-TIC not required.
- Reconstructed ion chromatogram(s) and quantitation report(s) or legible facsimile (GC/MS), labeled as in 8.3.2.3.2.5 above. Spectra not required.

8.3.2.4 Semivolatile Data

8.3.2.4.1 QC Summary

8.3.2.4.1.1 Surrogate Percent Recovery Summary (Form II-SV)

8.3.2.4.1.2 Control Matrix Spike Summary (Form III-SV)

8.3.2.4.1.3 Method Blank Summary (Form IV-SV)

(If more than a single form is necessary, forms must be arranged in chronological order by date of extraction of the blank.)

8.3.2.4.1.4 GC/MS Tuning and Mass Calibration (Form V-SV) (in chronological order; by instrument).

8.3.2.4.1.5 Internal Standard Area and RT Summary (Form VIII-SV) in chronological order, by instrument.

8.3.2.4.2 Sample Data

8.3.2.4.2.1 Sample data shall be arranged in packets with the Organic Analysis Data Sheet (Form I SV-1 and SV-2, including Form I SV-TIC), followed by the raw data for semivolatile samples. These sample packets should then be placed in increasing EPA sample number order, considering both letters and numbers in ordering samples. Phase units within a sample should be placed in order by phase unit suffix (i.e.; AB123-11, AB123-12, etc.).

8.3.2.4.2.2 TCL Results — Semivolatile Analysis Data Sheet (Form I SV-1, SV-2).

Tabulated results (identification and quantitation) of the specified target compounds (Exhibit C) of SOW OLM03.2. The validation and release of these results is authorized by a specific signed statement in the SDG Narrative (reference 8.3.2.1).

In the event that the Laboratory Manager cannot validate all data reported for each sample, the Laboratory Manager shall provide a detailed description of the problems associated with the sample in the SDG Narrative.

On Form I, the appropriate concentration units shall be µg/L or µg/Kg. No other units are acceptable. NOTE: Report analytical results to one significant figure if the value is less than 10; to two significant figures above 10.

8.3.2.4.2.3 Tentatively Identified Compounds (Form I SV-TIC).

This form must be included even if no compounds are found. If so, indicate this on the form by entering "0" in the field for "Number found".

Form I SV-TIC is the tabulated list of the highest probable match for up to 30 of the nonsurrogate organic compounds not listed in Exhibit C (TCL), including the CAS (Chemical Abstracts Registry) number, tentative identification and estimated concentration. For estimating concentration, assume a response factor of 1, and estimate the concentration by comparison of the compound peak height or total area count to the peak height or total area of the nearest internal standard free of interferences on the reconstructed ion chromatogram.

NOTE: The laboratory must be consistent (i.e., use peak height for all comparison or use total area count for all comparison).

8.3.2.4.2.4 Reconstructed total ion chromatograms (RIC) for each sample, sample extract, standard, blank, and spiked sample.

RICs must be normalized to the largest nonsolvent component, and must contain the following header information.

- EPA sample number
- Date and time of analysis
- GC/MS instrument ID
- Lab file ID

Internal standard and surrogate spiking compounds are to be labeled with the names of compounds, either directly out from the peak, or on a print-out of retention times if retention times are printed over the peak. If automated data system procedures are used for preliminary identification and/or quantification of the Target Compound List (TCL) compounds, the complete data system report must be included in all sample data packages, in addition to the

reconstructed ion chromatogram. The complete data system report shall include all of the information listed below. For laboratories which do not use the automated data system procedures, a laboratory "raw data sheet," containing the following information, must be included in the sample data package in addition to the chromatogram.

- EPA sample number
- Date and time of analysis
- RT of identified TCL compounds
- Ion used for quantitation with measured area
- Copy of area table from data system
- GC/MS instrument ID
- Lab file ID
- Analyst ID

8.3.2.4.2.5 For each sample, by each compound identified:

- Copies of raw spectra and copies of background-subtracted mass spectra of target compound listed in Exhibit C of SOW OLM03.2 (TCL) that are identified in the sample and corresponding background-subtracted TCL standard mass spectra. Spectra must be labeled with EPA sample number, lab file ID, date and time of analysis, and GC/MS instrument ID; compound names must be clearly marked on all spectra.

- Copies of mass spectra of non-surrogate organic compounds not listed in Exhibit C of SOW OLM03.2 (TCL) (Tentatively Identified Compounds) with associated best-match spectra (three best matches), labeled as in 8.3.2.4.2.5 above.

8.3.2.4.3 Standard Data

8.3.2.4.3.1 Initial Calibration Data (Form VI SV-1, SV-2) - in order by instrument, if more than one instrument used.

- Semivolatile standard(s) reconstructed ion chromatograms and quantitation reports (or legible facsimile) for the initial (three-point) calibration, labeled as in 8.3.2.4.2.4 above. Spectra are not required.
- All initial calibration data must be included, regardless of when it was performed and for which case. When more than one initial calibration is performed, the data must be put in chronological order, by instrument.
- EICPs displaying each manual integration.

8.3.2.4.3.2 Continuing Calibration (Form VII SV-1, SV-2) - in order by instrument, if more than one instrument used.

- Semivolatile standard(s) reconstructed ion chromatograms and quantitation reports (or legible facsimile) for all continuing (12 hour) calibrations, labeled as in 8.3.2.4.2.4 above. Spectra are not required.
- When more than one continuing calibration is performed, forms must be in chronological order, by instrument.
- EICPs displaying each manual integration.

8.3.2.4.4 Raw QC Data

8.3.2.4.4.1 DFTPP (for each 12-hour period, for each GC/MS system utilized)

- Bar graph spectrum, labeled as in 8.3.2.4.2.4 above.
- Mass listing, labeled as in 8.3.2.4.2.4 above.

- Reconstructed Total Ion Chromatogram, labeled as in 8.3.2.4.2.4 above.

8.3.2.4.4.2 Blank Data — in chronological order by extraction date. NOTE: This order is different from that used for samples.

- Tabulated results (Form I SV-1, SV-2)
- Tentatively Identified Compounds (Form I SV-TIC) —even if none found.
- Reconstructed ion chromatogram(s) and quantitation report(s) or legible facsimile (GC/MS), labeled as in 8.3.2.4.2.4 above.
- TCL spectra with lab generated standard, labeled as in 8.3.2.4.2.5 above. Data systems which are incapable of dual display shall provide spectra in order:
  - Raw TCL compound spectra
  - Enhanced or background subtracted spectra
  - Laboratory generated TCL standard spectra
- GC/MS library search spectra for Tentatively Identified Compounds (TIC), labeled as in 8.3.2.4.2.5 above.
- Quantitation/Calculation of Tentatively Identified Compound(s) (TIC) concentrations.

8.3.2.4.4.3 Matrix Spike and Duplicate Data

- Tabulated results (Form I) TCL compounds. Form 1 SV-TIC <u>not</u> required.
- Reconstructed ion chromatogram(s) and quantitation report(s) or legible facsimile (GC/MS), labeled as in 8.3.2.4.2.4 above. Spectra <u>not</u> required.

8.3.2.4.5 Raw GPC Data

8.3.2.5 Pesticide/PCB Data

8.3.2.5.1 QC Summary

8.3.2.5.1.1 Surrogate Recovery Summary (Form II PEST)

8.3.2.5.1.2 Pesticide Control Matrix Spike Recovery Summary (Form III PEST)

8.3.2.5.1.3 Pesticide Method Blank Summary (Form IV PEST)

(If more than a single form is necessary, forms must be arranged in chronological order by date of analysis of the blank.)

8.3.2.5.2 Sample Data

8.3.2.5.2.1 Sample data shall be arranged in packets with the Organic Analysis Data Sheet (Form I PEST), Followed by the raw data for samples. These sample packets should then be placed in increasing EPA sample number order, considering both letters and numbers in ordering samples.

8.3.2.5.2.2 TCL Results-Analysis Data Sheet (Form I).

Tabulated results (identification and quantitation) of the specified target compounds (Exhibit C) of SOW OLM03.2. The validation and release of these results is authorized by a specific, signed statement in the SDG Narrative (reference C.1). In the event that the Laboratory Manager cannot validate all data reported for each sample, the Laboratory Manager shall provide a detailed description of the problems associated with the sample in the SDG Narrative.

On Form I, the appropriate concentration units shall be µg/L or µg/Kg. No other units are acceptable.

NOTE: Report analytical results to two significant figures for all Pesticide/PCB samples.

8.3.2.5.2.3 Copies of Pesticide/PCB analysis chromatograms.

All chromatograms must be labeled with the following information:

- EPA sample number
- Volume injected (µl)
- Date and time of injection
- GC column identification (by stationary phase)
- GC instrument identification
- Positively identified compounds must be labeled with the names of compounds, either directly out from the peak, or on a print-out of retention times if retention times are printed over the peak.

8.3.2.5.2.4 Copies of Pesticide/PCB analysis chromatograms from second GC column confirmation. Chromatograms to be labeled as in 8.3.2.5.2.3 above.

8.3.2.5.2.5 GC Integration Report or data system printout.

8.3.2.5.2.6 Manual work sheets.

8.3.2.5.3 Standard Data

8.3.2.5.3.1 Form VI PEST-1, PEST-2, PEST-3, PEST-4, PEST-5, PEST-6, and PEST-7 — Initial Calibration Forms for all GC columns, all instruments, in chronological order by GC column and instrument.

8.3.2.5.3.2 Form VII PEST-1 and PEST-2 - Continuing Calibration Forms for all GC columns, EICPs displaying each manual integration.

8.3.2.5.3.3 Form VIII - Pesticide Analytical Sequence for all GC columns, EICPs displaying each manual integration.

8.3.2.5.3.4 Form IX: PEST-1— Pesticide Florisil cartridge check. PEST-2 — Pesticide GPC calibration.

8.3.2.5.3.5 Form X Pesticide Identification Summary for (PEST-1, PEST-2)

8.3.2.5.3.6 Pesticide/PCB standard chromatograms and data system printouts for all standards to include:

- All quantitation standards
- A copy of the computer reproduction or strip chart recorder output covering the 100 fold range

- All chromatograms are required to have the following:

  - Label all standard peaks for all individual compound either directly out from the peak or on the printout of retention times if retention times are printed over the peak.
  - Label the chromatogram for multicomponent standards. (i.e.; Arochlor 1242, Toxaphene)
  - List total ng injected for each standard.
  - Date and time of injection.
  - GC column identification (by stationary phase).
  - GC instrument identification.
  - Scaling Factor.

8.3.2.5.4 Raw QC Data

8.3.2.5.4.1 Blank data — arranged by type (i.e., method, instrument, sulfur cleanup), in chronological order by instrument. NOTE: This order is different from that used for samples.

- Tabulated results (Form I).
- Chromatogram(s) and data system printout(s) (GC) for each GC column and instrument used for analysis labeled as in 8.3.2.5.2.2 above.

8.3.2.5.4.2 Matrix Spike and Duplicate Data

- Tabulated results (Form I) TCL compounds.
- Chromatogram(s) and data system printout(s) (GC), labeled as in 8.3.2.5.2.2 above.

8.3.2.5.5 Raw GPC Data

8.4 GC/MS Tapes

8.4.1 The Contractor must store all raw and processed GC/MS data on magnetic tape, in appropriate instrument manufacturer's format. This tape must include data for samples, blanks, control matrix spikes, initial calibrations, continuing calibrations BFB and DFTPP, as well as all laboratory-generated spectral libraries and quantitation reports required to generate the data package. The Contractor shall maintain a written reference logbook of tape files to EPA sample number, calibration data, standards, blanks, and control matrix spikes. The logbook should include EPA sample numbers and standard and blank ID's identified by Case and Sample Delivery Group.

8.4.2 The Contractor is required to retain the GC/MS tapes for 365 days after data submission. During that time, the Contractor shall submit tapes and associated logbook pages within seven days after receipt of a written request from the Project Officer or the Sample Management Office.

9.0 CALCULATIONS. N/A

10.0 QUALITY ASSURANCE/QUALITY CONTROL. N/A

11.0 WASTE DISPOSAL. N/A

12.0 METHOD PERFORMANCE. N/A

13.0 REFERENCES. N/A

14.0 DEFINITIONS

14.1 Terminology

SDG Narrative:..... Portion of the data package that includes sample episode or SDG information, and descriptive documentation of any problems encountered n analyzing or processing.

Extra Data:..... Data classified as non-reportable, but stored by the laboratory with the pertinent episode information for reference purposes.

14.2 Acronyms

CLP: ..... Contract Laboratory Program

SDG: ..... Sample Delivery Group

EPA: ..... Environmental Protection Agency

CSF: ..... Complete SDG File (Original)

LIMS: ..... Laboratory Information Management System

TCL: ..... Target Compound List

TIC: ..... Tentatively Identified Compounds

15.0 ATTACHMENTS

Attachment 1: ..... Case Packet Form (In-House)
Attachment 2: ..... Case Status Form
Attachment 3: ..... LIMS Generated
Attachment 4: ..... SDG Narrative
Attachment 5: ..... Sample Delivery Group
Attachment 6: ..... Client Sample Traffic Report
Attachment 7: ..... Figures for Form I's
Attachment 8: ..... Figures for Form II's
Attachment 9: ..... Figures for Form III's
Attachment 10: ..... Figures for Form IV's
Attachment 11: ..... Figures for Form V's
Attachment 12: ..... Figures for Form VI's
Attachment 13: ..... Figures for Form VII's
Attachment 14: ..... Figures for Form VIII's
Attachment 15: ..... Figures for Form IX's
Attachment 16: ..... Figures for Form X's

CASE PACKET FORM

SDG: _____

CASE NO.: _____

RECEIVED: _____

DUE DATE: _____

MATRIX: _____

FRACTION: _____

NOTE: ***All the figures throughout this SOP have been reduced in size for this document. All are actually 8½ x 11in.***

Attachment 1: Case Packet Form (in-house)

VOA CASE STATUS FORM

Date: _____ CLP NONCLP

Client/Case: _____ MS_____ # of Samples_____

Data pkg. Forms Only

SDG/Episode: _____ Episode #'s: _____

Lab Code: SWOK

Case written up by: _____ AIR / ALL WATERS / ALL SOILS / MIXED SAMPLES / GC SAMPLES

pH Form: _____

% Moisture Sheets: _____ Date Due: _____

TCLP Extraction Sheets: _____ _____th day

Clerks Notes:

Data Reviewer Notes:

Attachment 2: Case Status Form (Orange paper used). (Used for Volatiles Only)

SOUTHWEST LABORATORY OF OKLAHOMA, INC.
1700 W. ALBANY SUITE C
BROKEN ARROW, OK 74012-1421

Date: 10/18/97
Episode: 31304
Client: EPA
Project: 35734

***FILE LOG-IN RECORD *

Attachment 3: LIMS Generated

Attachment 4
SDG Narrative

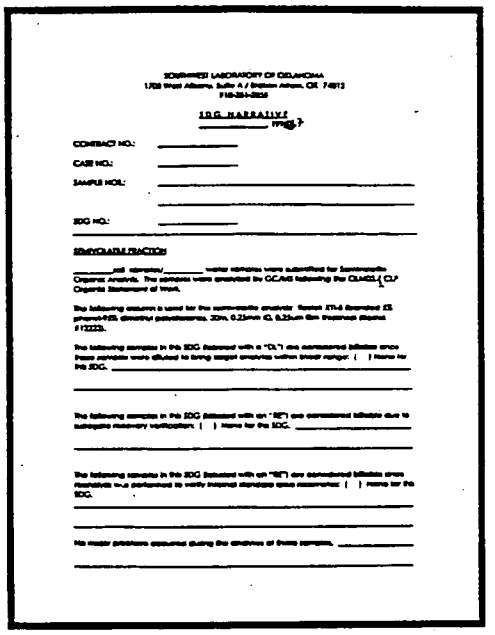


Figure 1

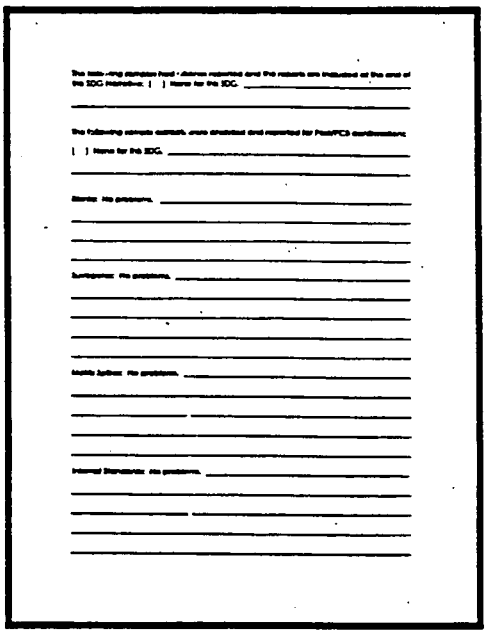


Figure 2

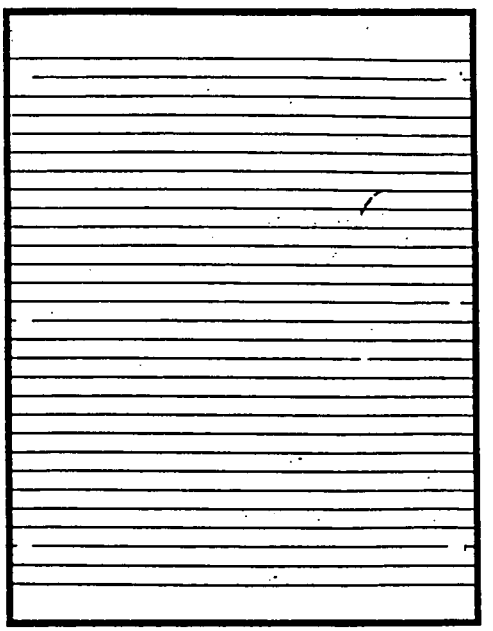
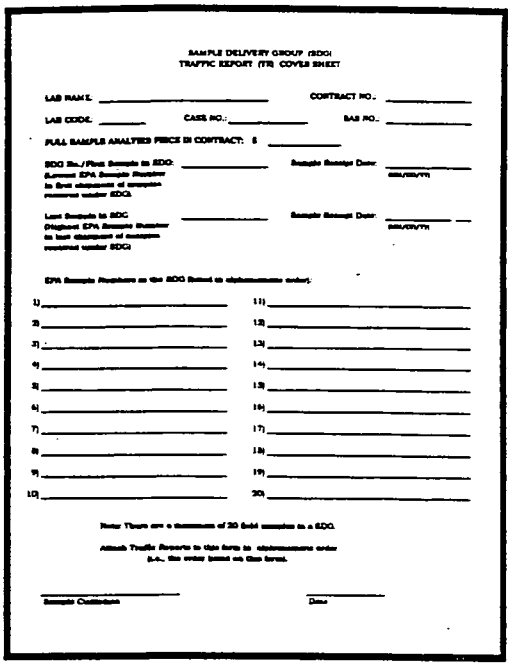


Figure 3



SAMPLE DELIVERY GROUP (SDG)
TRAFFIC REPORT (TR) COVER SHEET

LAB NAME: _____ CONTRACT NO.: _____

LAB CODE: _____ CASE NO.: _____ SAS NO.: _____

FULL SAMPLE ANALYSIS PRICE IN CONTRACT: $ _____

SDG No./First Sample in SDG: _____ Sample Receipt Date: _____
(Lowest EPA Sample Number in first shipment of samples received under SDG) MM/DD/YY

Last Sample in SDG: _____ Sample Receipt Date: _____
(Highest EPA Sample Number in last shipment of samples received under SDG) MM/DD/YY

EPA Sample Numbers in the SDG listed in alphanumeric order:

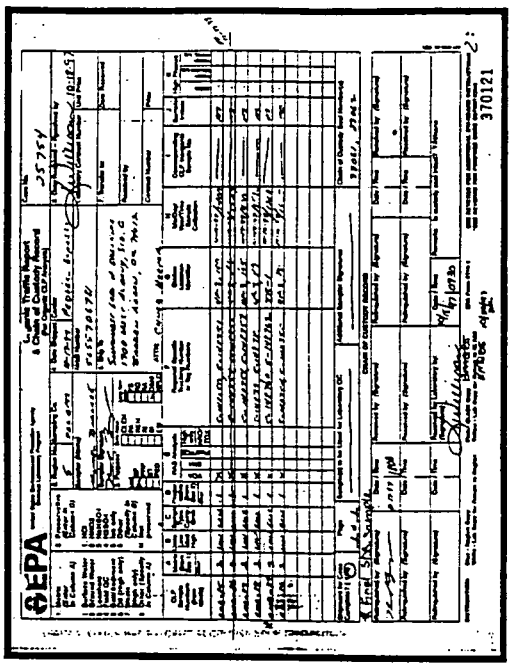
1) _____	11) _____
2) _____	12) _____
3) _____	13) _____
4) _____	14) _____
5) _____	15) _____
6) _____	16) _____
7) _____	17) _____
8) _____	18) _____
9) _____	19) _____
10) _____	20) _____

Note: There are a maximum of 20 field samples in a SDG.

Attach Traffic Reports to this form in alphanumeric order (i.e., the order listed on this form).

_____ Sample Custodian _____ Date

Attachment 5: Sample Delivery Group (SDG Traffic Report (TR) Cover Sheet



Attachment 6: Client Sample Traffic Report

Attachment 7

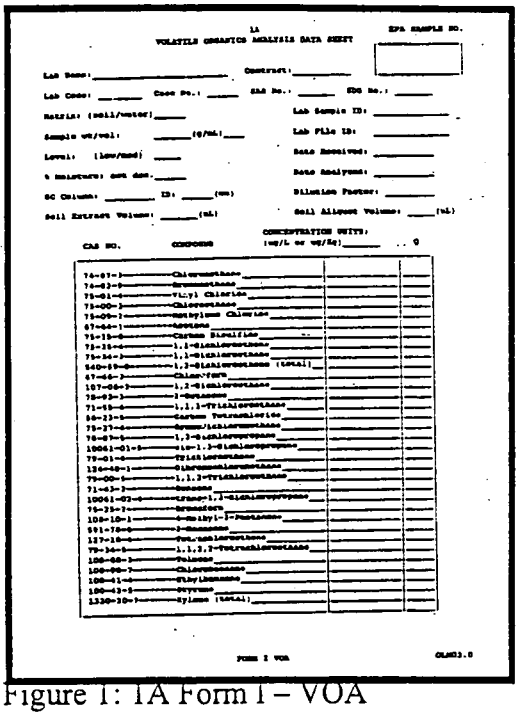


Figure 1: 1A Form I – VOA

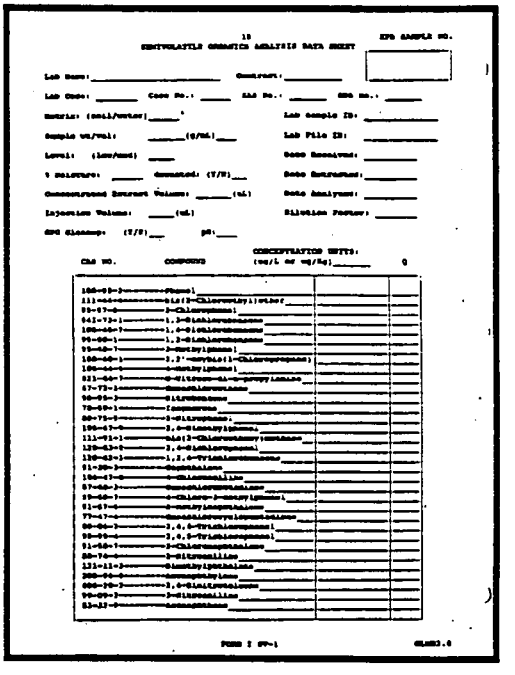


Figure 2: 1B Form I SV-1

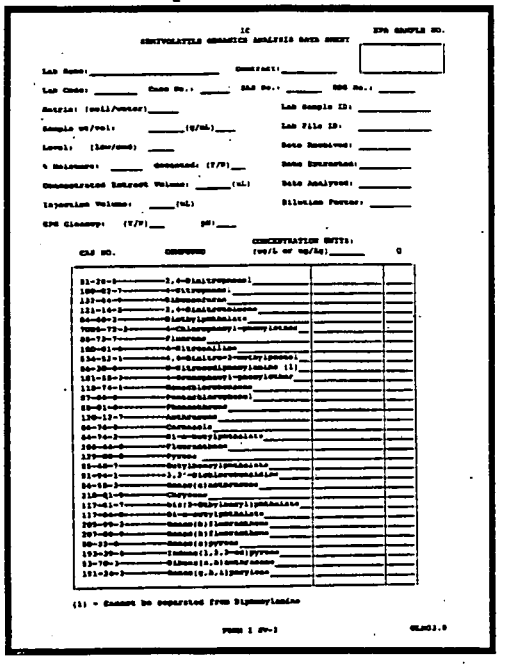


Figure 3: 1C Form I SV-2

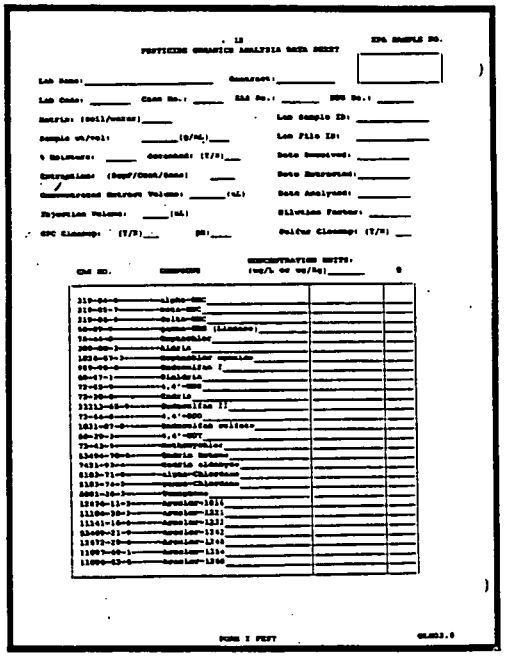


Figure 4: 1D Form I Pest

Attachment 7 (Continued)

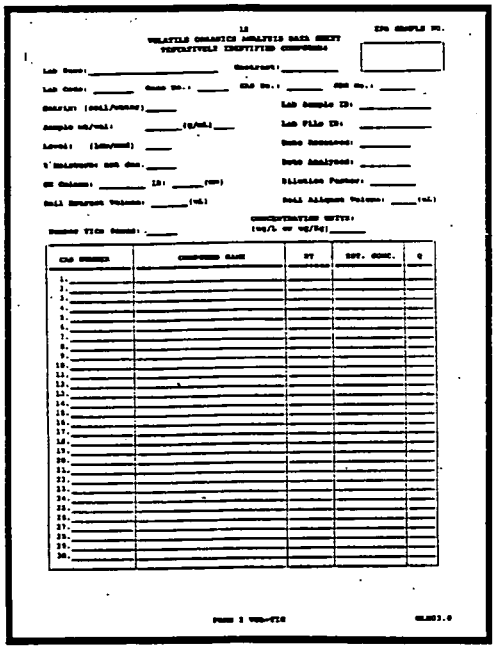


Figure 5: 1E Form I VOA-TIC

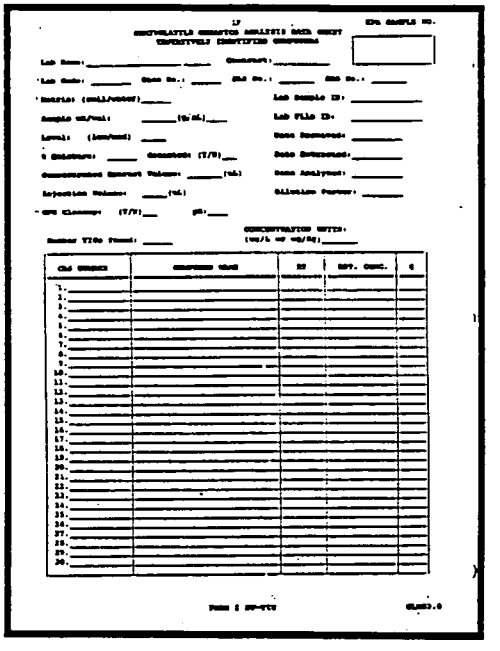


Figure 6: 1F Form I SV-TIC

Attachment 8

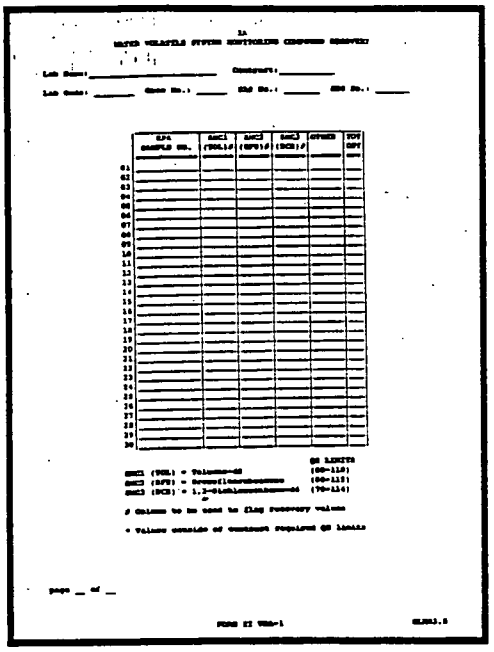


Figure 1: 2A Form II VOA-1

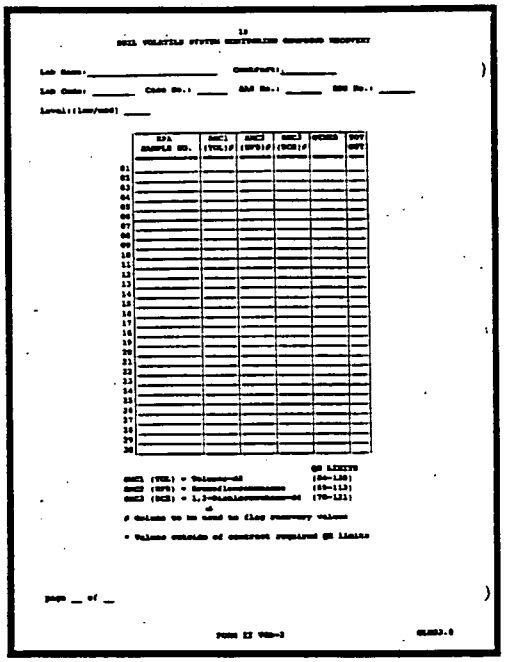


Figure 2: 2B Form II VOA-2

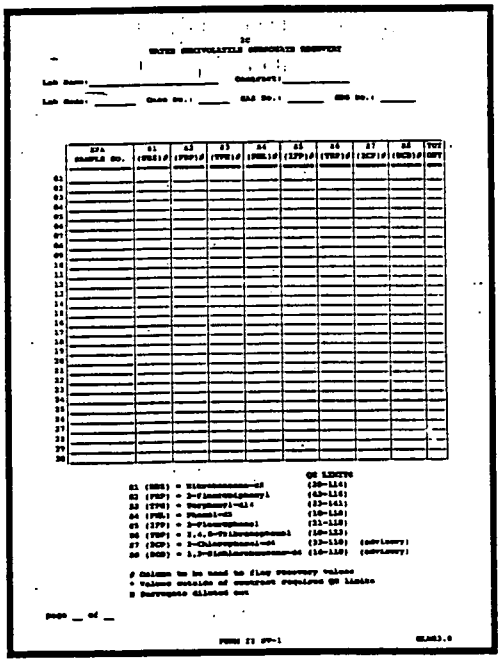


Figure 3: 2C Form II SV-1

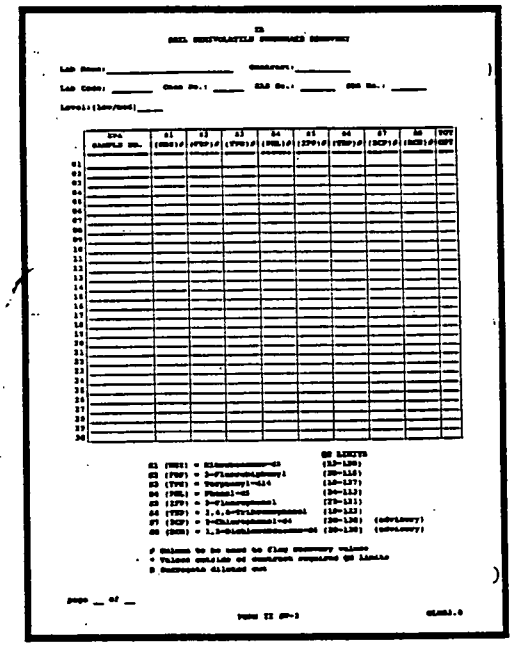


Figure 4: 2D Form II SV-2

Attachment 8 (Continued)

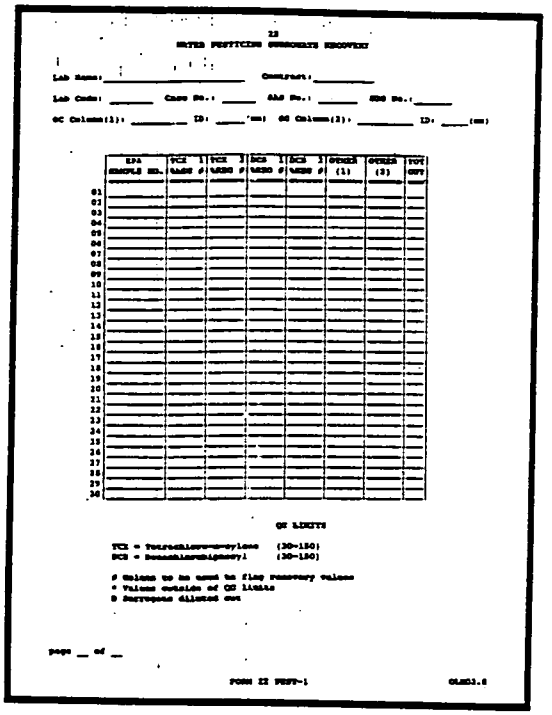


Figure 5: 2E Form II PEST-1

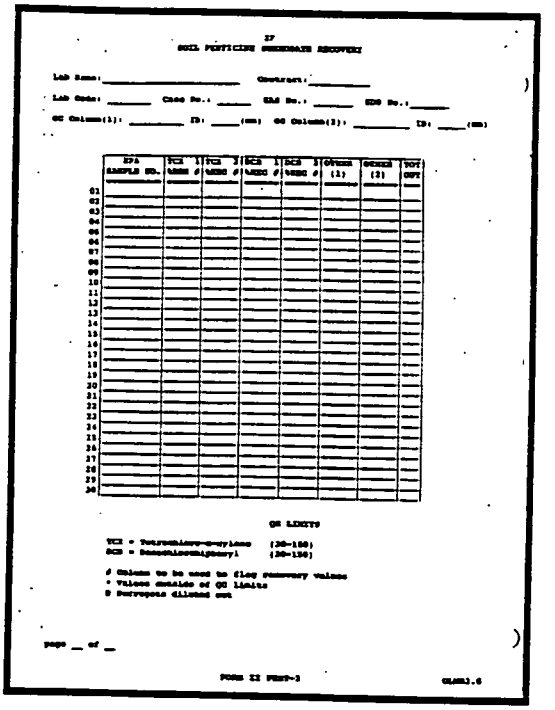


Figure 6: 2F Form II PEST-2

Attachment 9

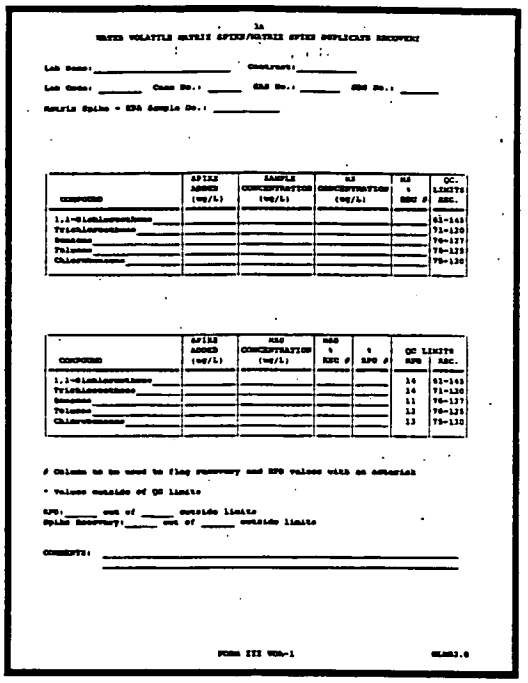


Figure 1: 3A Form III VOA-1

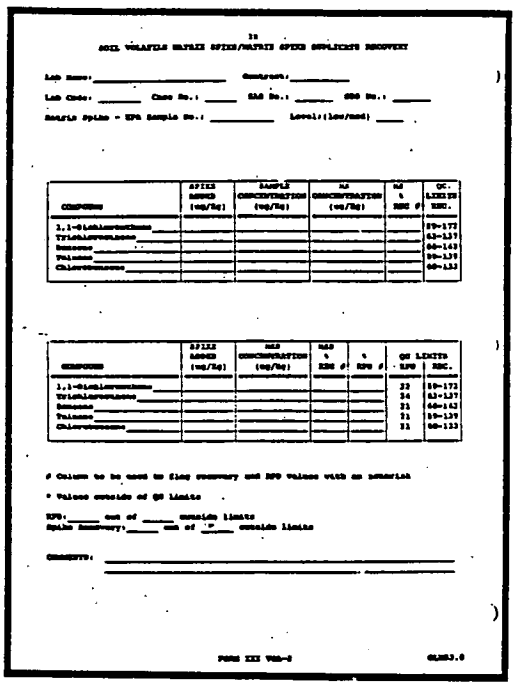


Figure 2: 3B Form III VOA-2

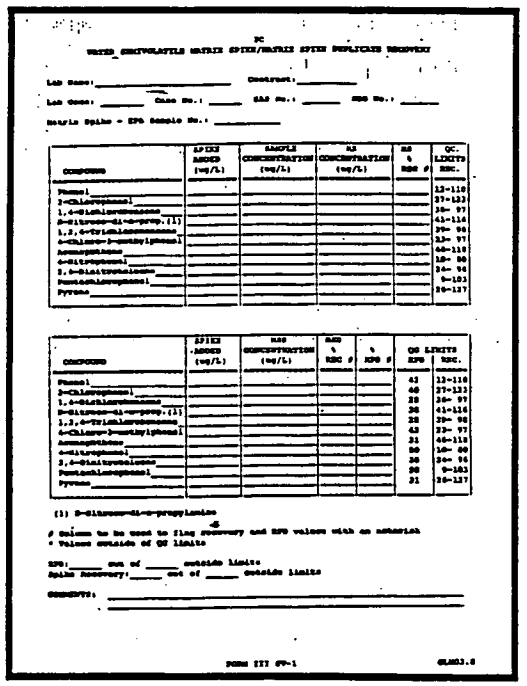


Figure 3: 3C Form III SV-1

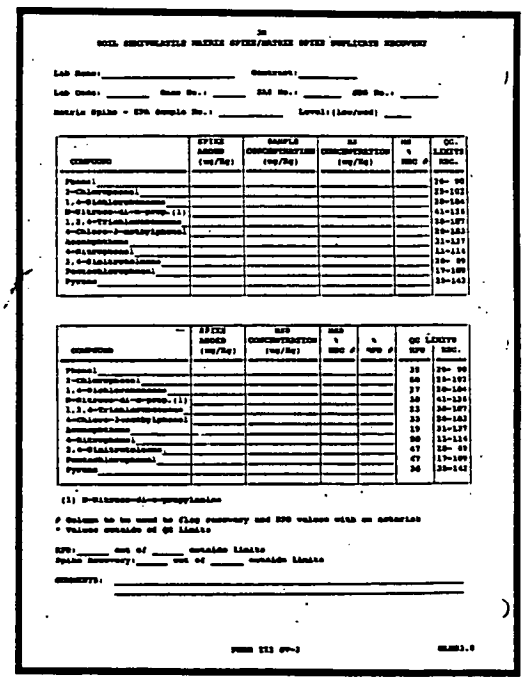


Figure 4: 3D Form III SV-2

Attachment 9 (Continued)

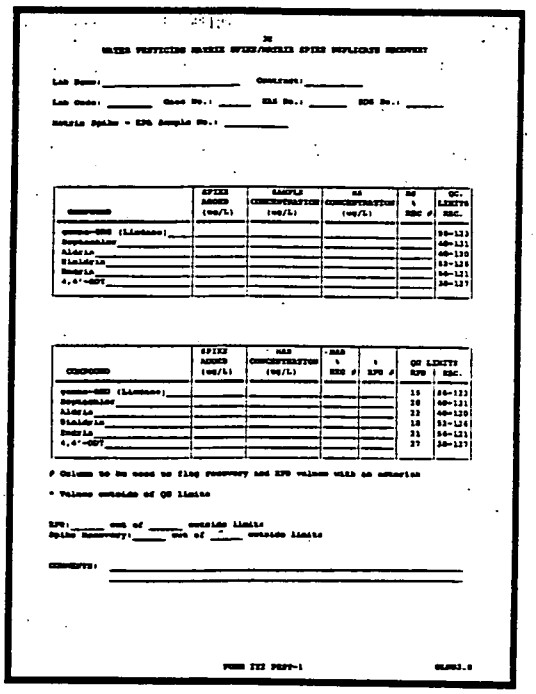


Figure 5: 3E Form III PEST-1

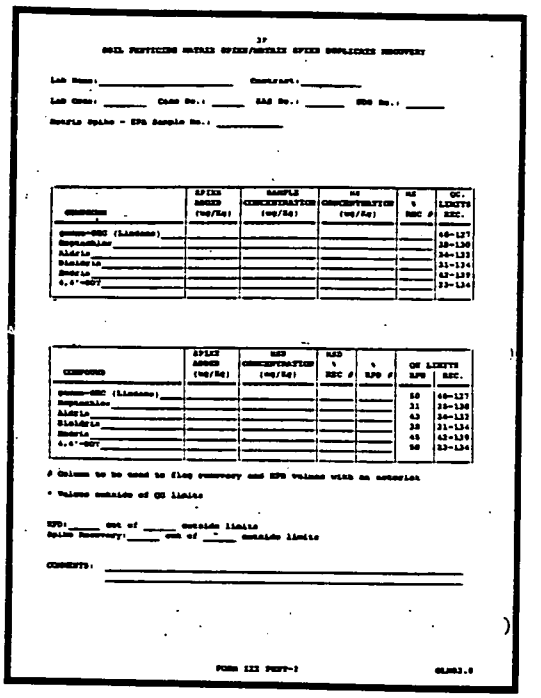


Figure 6: 3F Form III PEST-2

Attachment 10

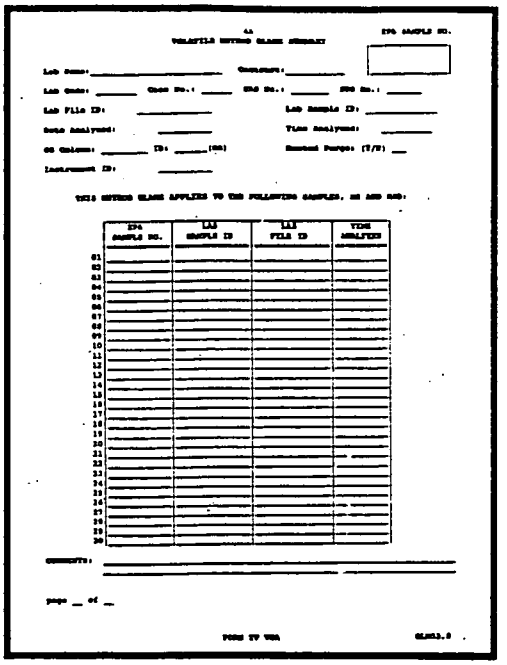


Figure 1: 4A Form IV VOA

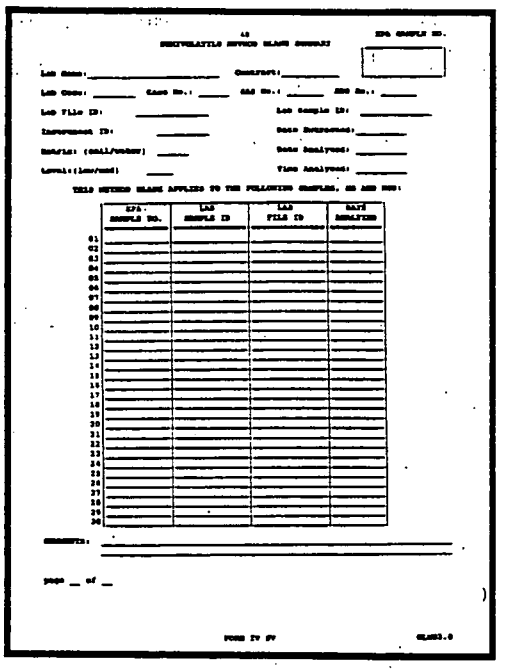


Figure 2: 4B Form IV SV

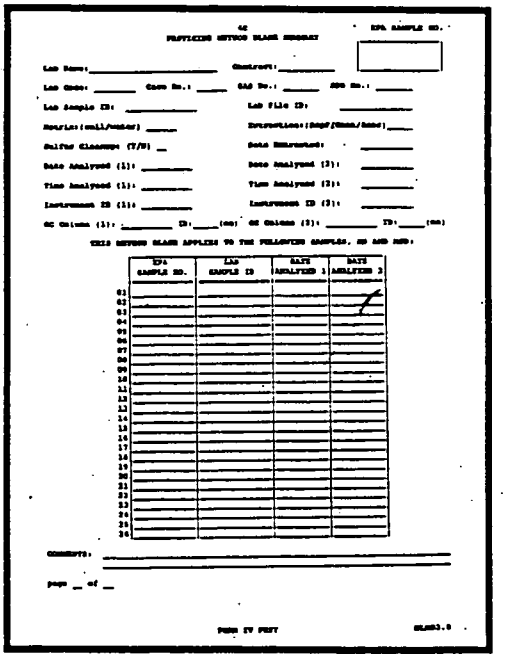


Figure 3: 4C Form IV PEST

Attachment 11

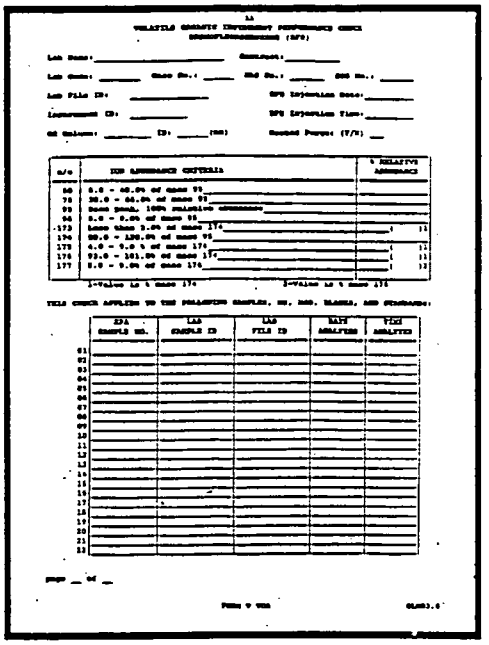


Figure 1: 5A Form V VOA

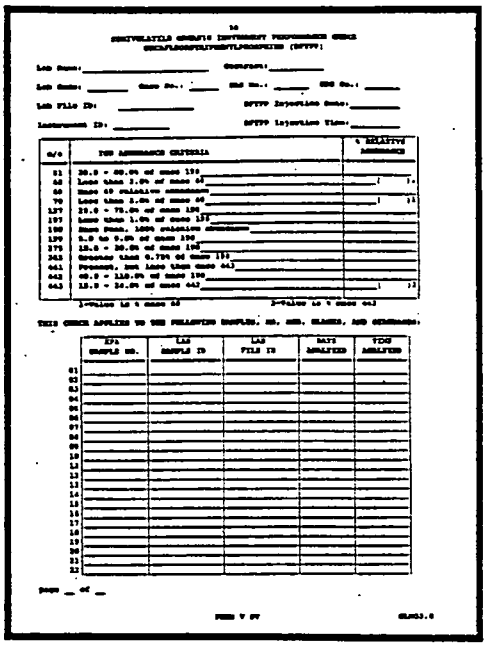


Figure 2: 5B Form V SV

Attachment 12

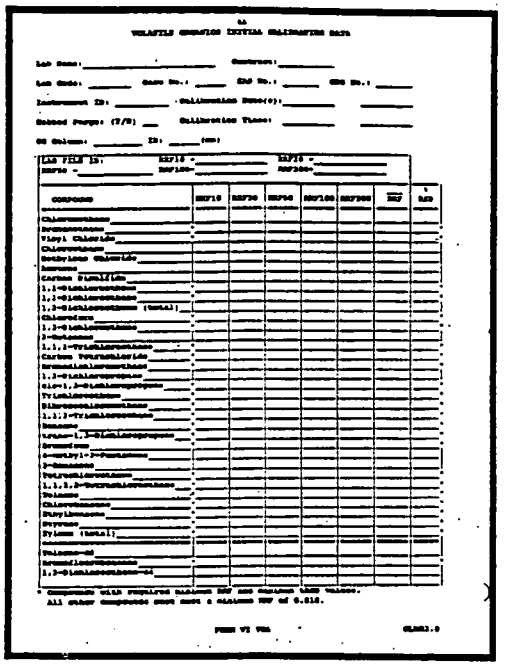


Figure 1: 6A Form VI VOA

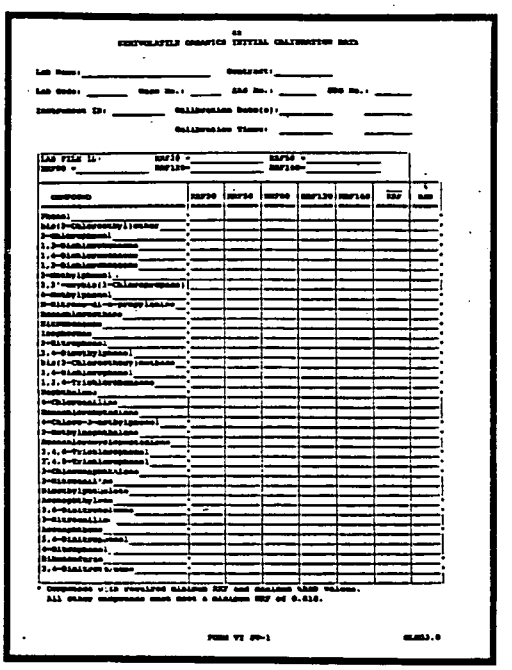


Figure 2: 6B Form VI SV-1

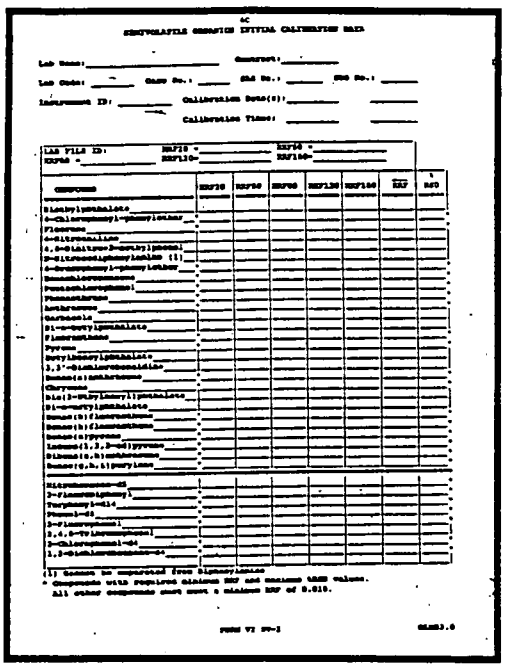


Figure 3: 6C Form VI SV-2

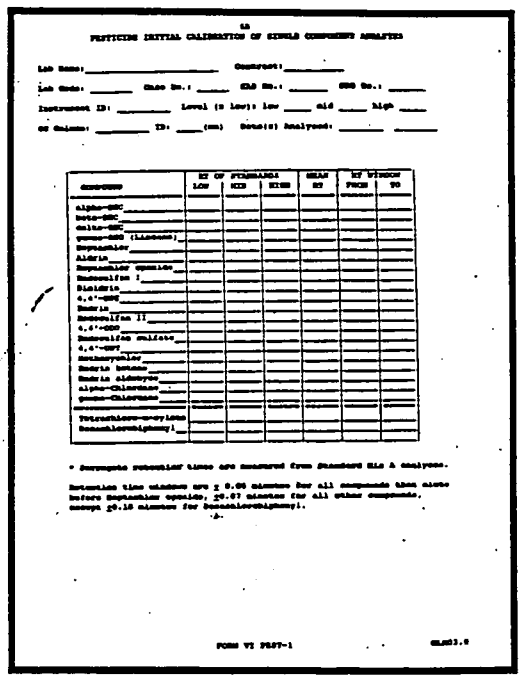


Figure 4: 6D Form VI PEST-1

Attachment 12 (Continued)

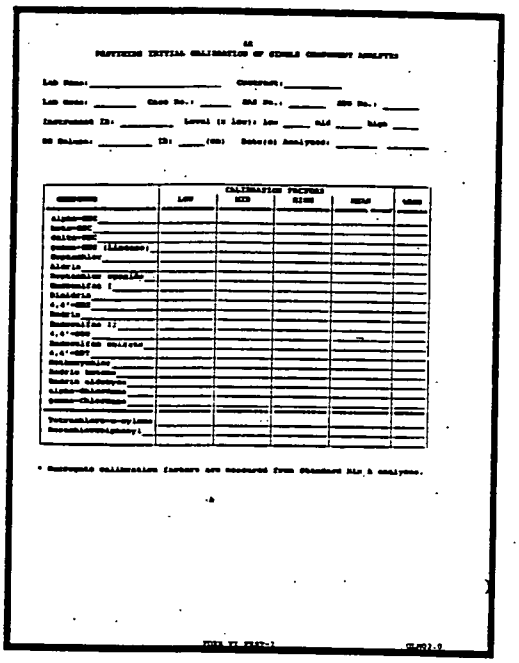


Figure 5: 6E Form VI PEST-2

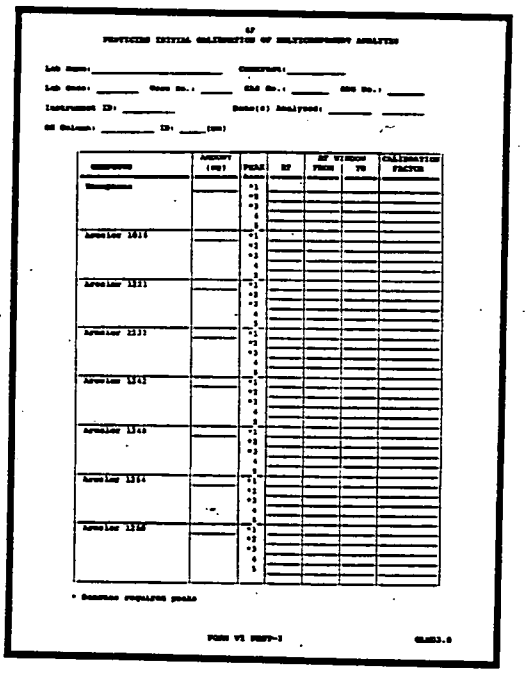


Figure 6: 6F Form VI PEST-3

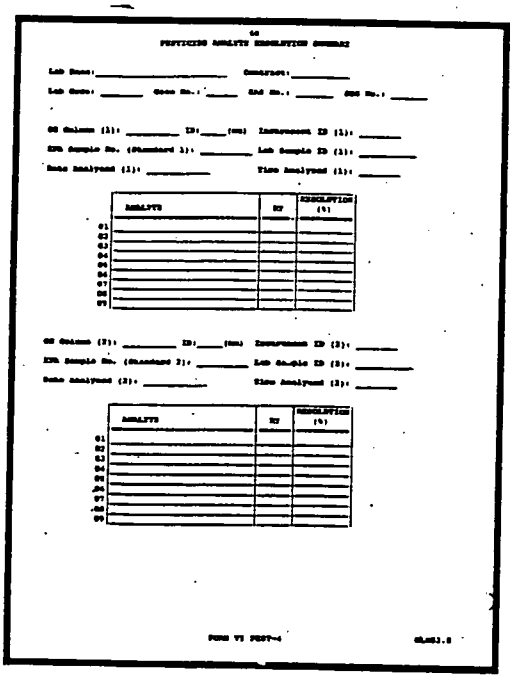


Figure 7: 6G Form VI PEST-4

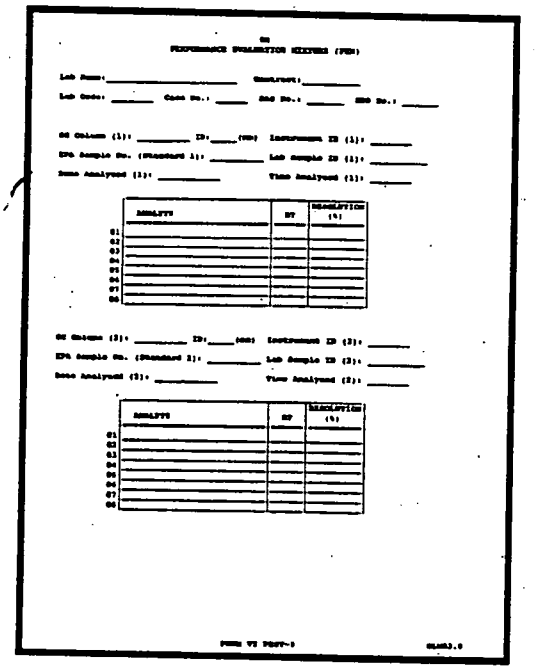


Figure 8: 6H Form VI PEST-5

Attachment 12 (Continued)

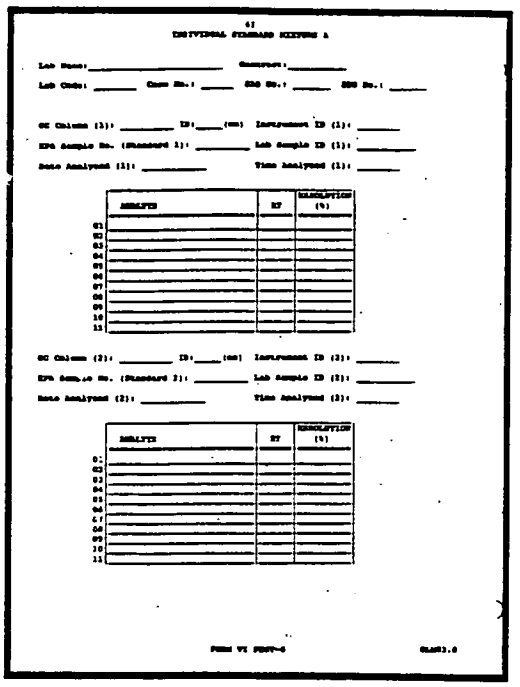


Figure 9: 6I Form VI PEST-6

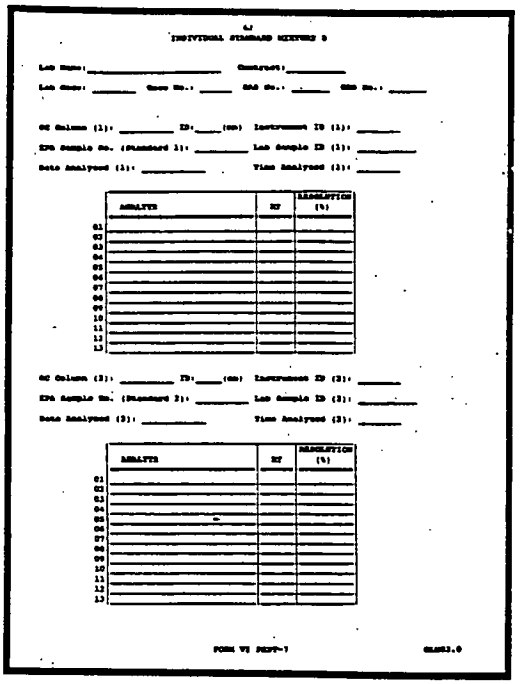


Figure 10: 6J Form VI PEST-7

Attachment 13

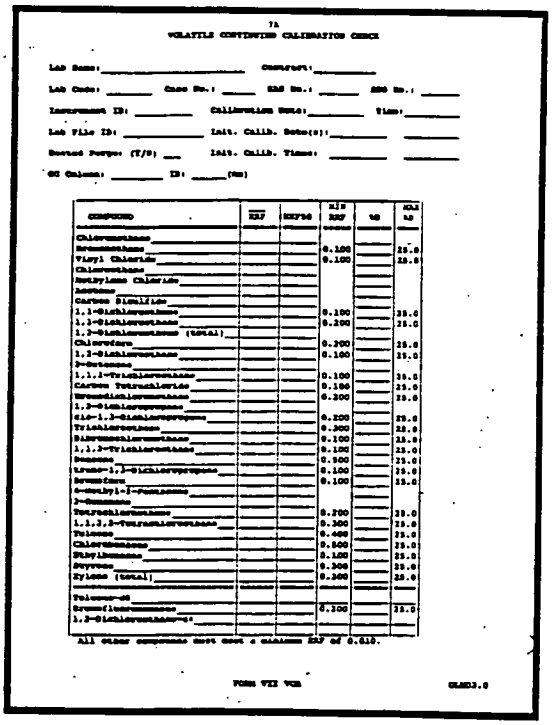


Figure 1: 7A Form VII VOA

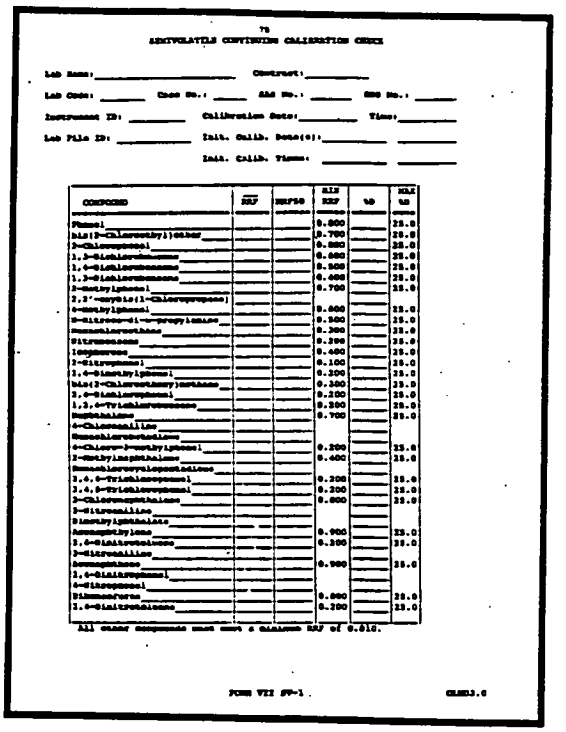


Figure 2: 7B Form VII SV-1

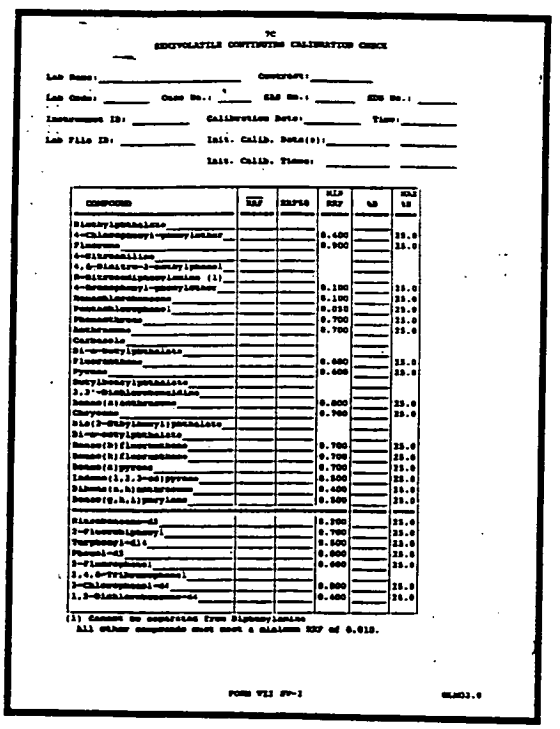


Figure 3: 7C Form VII SV-2

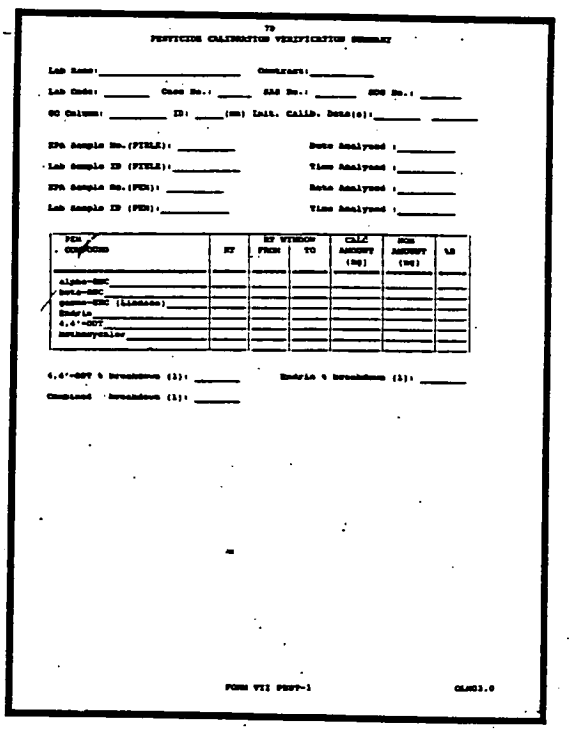
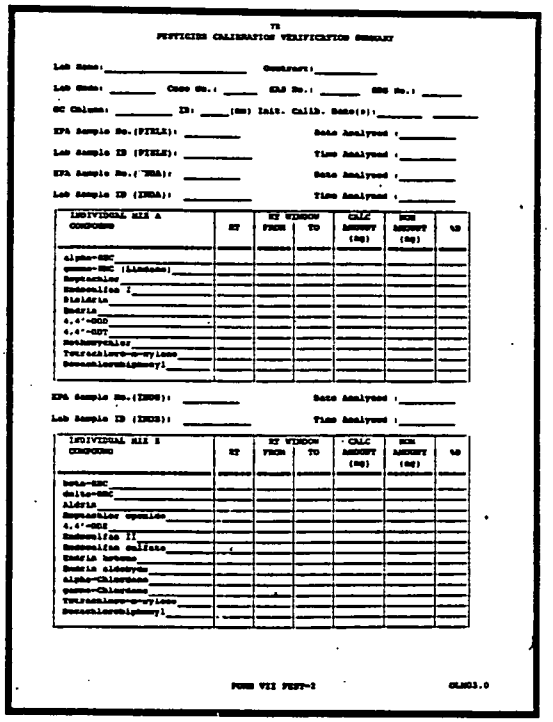


Figure 4: 7D Form VII PEST-1

Attachment 13 (Continued)



7E
PESTICIDE CALIBRATION VERIFICATION SUMMARY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

GC Column: _____ ID: ____(mm) Init. Calib. Date(s): _____ _____

EPA Sample No.(PTSL1): _____ Date Analyzed 1: _____

Lab Sample ID (PTSL1): _____ Time Analyzed 1: _____

EPA Sample No.(TMA): _____ Date Analyzed 1: _____

Lab Sample ID (IMBA): _____ Time Analyzed 1: _____

INDIVIDUAL MIX A COMPOUND	RT	RT WINDOW FROM	RT WINDOW TO	CALC AMOUNT (ng)	RCN AMOUNT (ng)	%D
alpha-BHC						
gamma-BHC (Lindane)						
Heptachlor						
Endosulfan I						
Dieldrin						
Endrin						
4,4'-DDD						
4,4'-DDT						
Methoxychlor						
Tetrachloro-m-xylene						
Decachlorobiphenyl						

EPA Sample No.(IMB8): _____ Date Analyzed 1: _____

Lab Sample ID (IMB8): _____ Time Analyzed 1: _____

INDIVIDUAL MIX B COMPOUND	RT	RT WINDOW FROM	RT WINDOW TO	CALC AMOUNT (ng)	RCN AMOUNT (ng)	%D
beta-BHC						
delta-BHC						
Aldrin						
Heptachlor epoxide						
4,4'-DDE						
Endosulfan II						
Endosulfan sulfate						
Endrin ketone						
Endrin aldehyde						
alpha-Chlordane						
gamma-Chlordane						
Tetrachloro-p-xylene						
Decachlorobiphenyl						

FORM VII PEST-2 SLM03.0

Figure 5: 7E Form VII Pest-2

Attachment 14

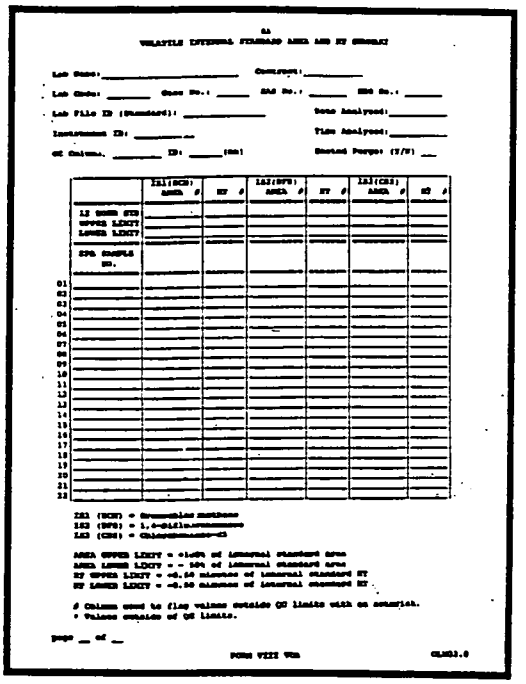


Figure 1: 8A Form VIII VOA

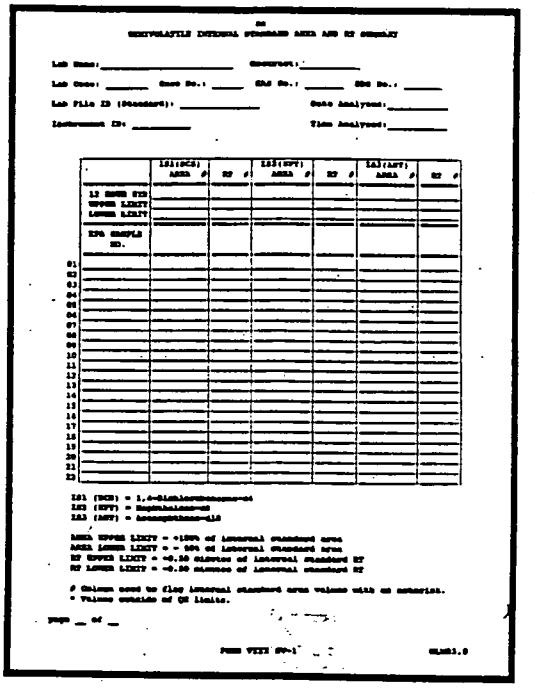


Figure 2: 8B Form VIII SV-1

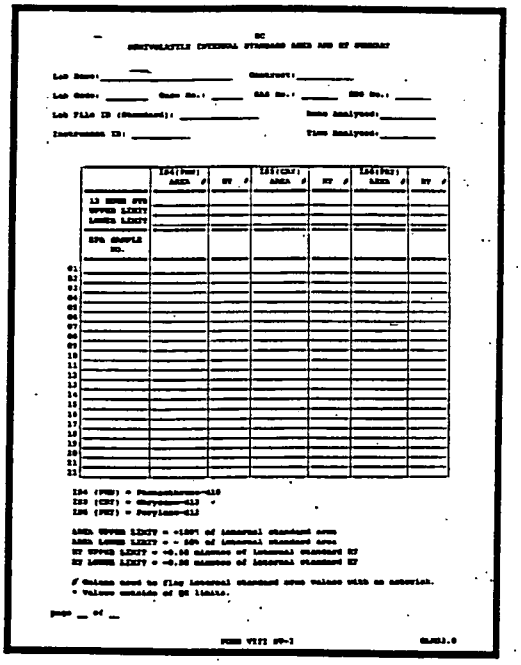


Figure 3: 8C Form VIII SV-2

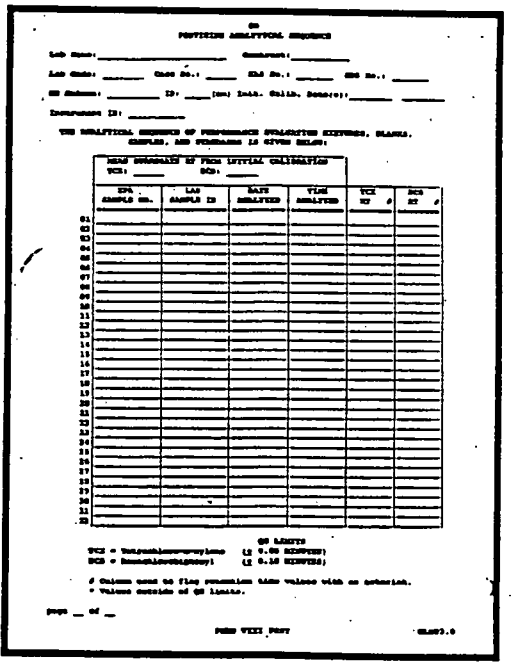


Figure 4: 8D Form VIII PEST

Attachment 15

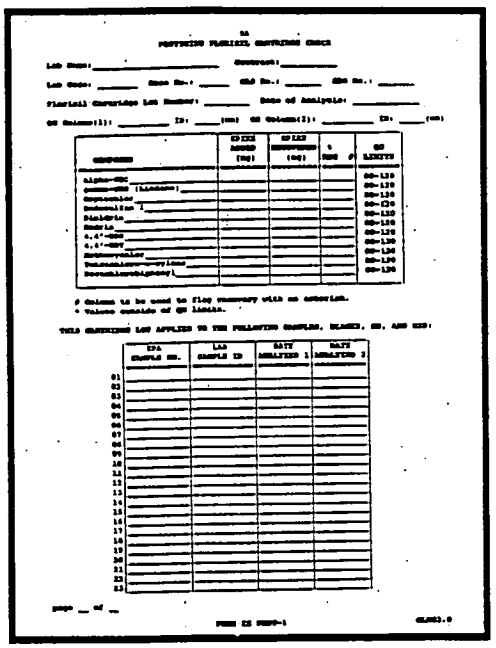


Figure 1: 9A Form IX PEST-1

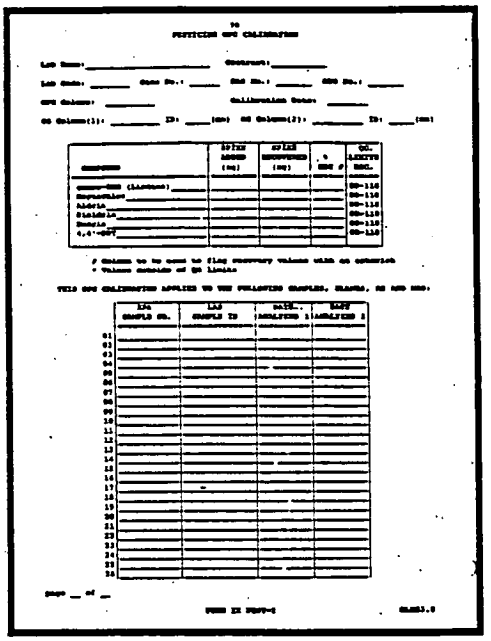


Figure 2: 9B Form IX PEST-2

Attachment 16

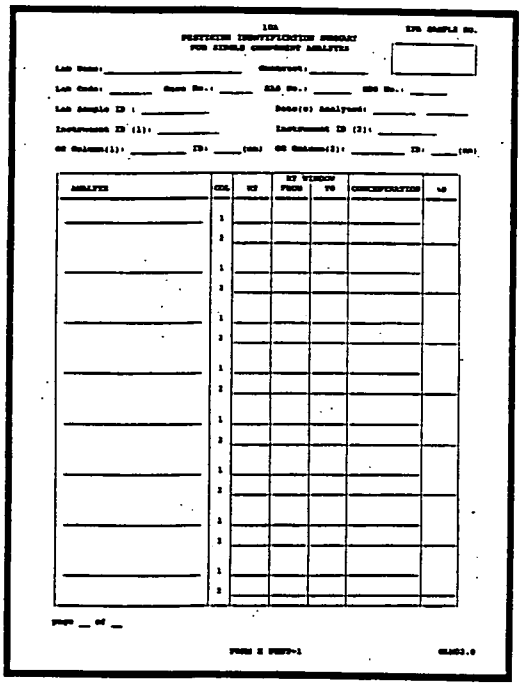


Figure 1: 10A Form X PEST-1

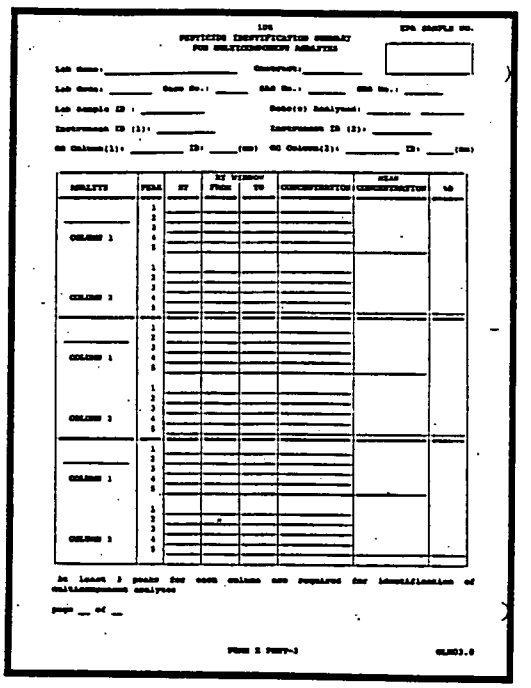


Figure 2: 10B Form X PEST-2

SOUTHWEST LABORATORY OF OKLAHOMA, INC. and
AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.

TM

Standard Operating Procedure

Analysis of Semivolatile Organics by SW846-8270C

Document No.: SWL-OS-500

Rev No. / Date: Rev. 6.0 — 07/07/00

OFFICIAL COPY RED STAMP INDICATES ORIGINAL

APPROVALS	DATES
Procedure Prepared By	Date 07-20-00
Program Manager or Section Supervisor	Date 7-20-00
Laboratory Safety Officer	Date 7/21/2000
Laboratory QA/QC Officer	Date 7/20/00

(Effective Date is 5 calendar days after the last signature above - QA/QC Officer)

Document Status

[ ] Controlled DC No. _____ Issued to: _____ Date: _____ QA: _____

[✓] Official Copy OC No.: 2431 Issued to: Linda Karamerich Date: 11/14/00 QA: _____

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ANALYSIS OF SEMIVOLATILE ORGANICS BY SW846-8270C

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1.0 SCOPE AND APPLICATION

1.1 Method 8270C (GC/MS full scan mode) is used to determine the concentration of various organic compounds in extracts prepared from all types of solid waste matrices, soils, oils, and ground/waste waters.

This method is used to quantitate organic samples that are soluble in methylene chloride and are capable of being eluted without derivatization as sharp peaks from a gas chromatographic fused-silica column (Restek XTI-5 or equivalent.)

1.2 Practical Quantitation Limits (PQL) for water analytes under this method have a typical range of 10 ug/L to 25 ug/L, and for solids 330 ug/Kg to 830 ug/Kg. These limits are highly matrix dependent and PQLs are proportionately higher for sample extracts that require cleanup or dilutions to avoid saturation of the detector. A list of the normal PQLs and compounds performed by this method is given in Table 10.

1.3 Modifications to the method are as per client specified analyte lists (i.e. Appendix 9, TCLP, Acid Extractables, Base-Neutral Extractables, Skinners, etc.).

2.0 METHOD SUMMARY

2.1 Method — This SOP (SWL-OS-500) is based on SW846-8270C, Revision 3, 1997, and has been written into a procedure for Southwest Laboratory of Oklahoma, Inc. This SOP is used for analyses of the following SWLO Test Codes: MS500, MS510, MS530, MS540, MS550, MS599, MS505, MS507.

2.2 Procedure — A measured volume of aqueous liquid or weight of solid sample is extracted with methylene chloride. For water samples the pH is adjusted if needed to greater than 11 for the initial extraction process; and then again at less than 2. The methylene chloride extract is dried and concentrated to a volume of 1.0 mL. For soil samples, the sample is dried with sodium sulfate and then sonicated with a 50-50 acetone-methylene chloride solvent and concentrated to a final extract of 1 mL. Chromatographic conditions are described which permit the separation and measurement of the parameters in the extract. Qualitative identification is performed using the retention time and the relative abundance of one to three characteristic ions. Quantitative analysis is performed by 5-point calibration and continuing calibration checks.

2.3 Extraction QC — With each batch of 20 samples or less, a blank, a laboratory control spike and a matrix/matrix spike duplicate (if sufficient sample is sent) must be prepared and analyzed.

3.0 HEALTH AND SAFETY

3.1 All samples and standards received by the laboratory should be considered hazardous; thus extreme care must be taken when handling. Care should be taken to ensure exposure by contact or inhalation is limited. All samples are to be handled in accordance with SOP SWL-GA-111 "*Laboratory Safety Plan*", and SWL-GA-114 "*Hazardous Waste Management Plan*".

3.1.1 Safety glasses and gloves (Laytex Powder Free) and lab coats should be utilized when prepping samples for analysis.

3.1.2 Fume Hoods, and/or respirators with organic vapor cartridges should be used for extremely loaded samples.

3.2 MSDS sheets are kept in the bench area for quick reference for all standards used in each department.

3.3 Carbon Traps are to be on all GC/MS instruments on the vent outlets to filter solvent and sample vapors being discharged.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

4.1 Containers

4.1.1 All samples are received from the extractions department in 1, 5, 10mL Borosilicate glass vials with black phenolic caps and Teflon lined septa.

4.1.2 All samples received by the extraction department are grouped in episodes and kept in sample vial file boxes.

4.1.3 An aliquot of each sample is transferred to a 1.8mL glass crimp top vial with a 250uL insert and sealed with an aluminum/Teflon/rubber septa cap for analysis.

4.2 Handling and Storage

4.2.1 The extraction personnel delivering the samples with extraction logs (Attachment 4) log all samples and QC in the Extract Receipt Log Attachment (see Attachment 5).

4.2.2 Once received from extractions the samples and QC are then logged in and checked off the extract receipt log by the GC/MS sample custodian.

4.2.3 All samples and QC are then placed in vial files in numerical order by episode and sample number, and stored in a refrigeration unit that is monitored daily with a NIST calibrated mercury thermometer and kept between 1°C to 4.4°C.

4.2.4 All standards used for analysis are received, handled and stored in accordance to the Laboratory's Standards SOP and Chemical Hygiene Plan.

5.0 INTERFERENCE AND POTENTIAL PROBLEMS — Interference and Potential Problems may be caused from the following:

5.1 Phthalate contamination from improper use of or type of gloves and other plastics.

5.2 Matrix contamination from high levels of non-target compounds found in sample.

5.3 Power spikes or surges from the electrical supply to the instrument

5.4 Dirty syringe on autosampler. Syringe should be checked daily for signs of wear.

5.5 Dirty, contaminated, or empty wash vials on autosampler.

5.5.1 Wash vials should be rinsed and refilled daily with clean solvent.

5.5.2 Wash solvent waste vials should be emptied daily

5.6 Insufficient disk space on data system

5.7 Improper capping of sample vials may cause evaporation of sample.

5.8 Failure of refrigeration units.

6.0 EQUIPMENT/APPARATUS

6.1 The GC/Mass Spectrometer (HP 5970, 71, 72, or 73, or equivalent, Quadropole MS) is capable of scanning from 35 to 500 amu every 1 second or less, utilizing 70 volts (nominal) electron energy in the electron impact ionization mode and producing a mass spectrum which meets all required criteria when 50ng of decafluorotriphenylphosphine (DFTPP) is injected through the GC inlet.

6.2 HP 5890 and HP 5890 Series II gas chromatographs in conjunction with the above mentioned GC/Mass Spectrometer.

- The recommended GC operating conditions to be used are as follows:

Initial Column Temperature Hold	—	40°C for 1 minute
Column Temperature Program	—	18°C/min. to 100°C
		15°C/min. to 325°C
		Hold 2.5 min.
		40°C/min. to 350°C
Injector Temperature	—	280°C
Transfer Line Temperature	—	280°C
Source Temperature	—	According to manufacturer's spec's.

NOTE: These conditions will vary depending on column and instrument.

6.3 HP7673 autosamplers with 100 vial capacity used for sample introduction into the HP5890/HP5970, 71, 72, 73 system.

6.4 Column — 30m x 0.25mm ID (or 0.32mm) bonded-phase silicone coated fused silica capillary column (Restek XTI-5 or equivalent.) A film thickness of 0.25 microns is used. A film thickness of 1.0 microns is recommended if a large loading capacity is needed.

6.5 Personal Computers equipped with ChemStation Software (HPChem) for the aquisition of sample data.

6.6 Computers equipped with Enviroquant software for the processing and review of acquired data.

6.7 HP LaserJet 3SIII/4SI printers for data hardcopies.

7.0 REAGENTS

7.1 Methylene Chloride — HPLC or Pesticide grade

7.1.1 Samples brought to final volume in Methylene Chloride for semi-volatile analysis.

7.1.2 Used as dilution solvent when dilutions are required on a sample.

7.1.3 Main solvent used in calibration standards and internal standard.

7.2 Initial Calibration Standards (See Table 11) — EPA traceable standards. All standards stored in original containers under temperature requirements as dictated by manufacture, (ambient or refrigerate). Once opened all standards are placed in amber borosilicate glass vials sealed with Teflon lined caps, and stored at -10°C to -20°C. All standards prepared in accordance with the Laboratory's Standards Preparation SOP.

NOTE: Equivalent EPA traceable standard by other manufacturers may be substituted.

7.2.1 S.V. Calibration Mix #1 (Restek)

7.2.2 S.V. Calibration Mix #2 (Restek)

7.2.3 S.V. Calibration Mix #3 (Restek)

7.2.4 S.V. Calibration Mix #4 (Restek)

7.2.5 S.V. Calibration Mix #5 (Restek)

7.2.6 S.V. Calibration Mix #7 (Restek)

7.2.7 Acid Surrogate Mix (Restek)

7.2.8 Base/Neutral Surrogate Mix (Restek)

7.2.9 Benzidines Mix (NSI)

7.2.10 Benzoic Acid (NSI)

7.2.11 Pyridine Stock (Chem. Service)

7.2.12 2,3,4,6-Tetrachlorophenol Stock (Chem. Service)

7.2.13 1,2,4,5-Tetrachlorobenzine Stock (Chem. Service)

7.2.14 Aniline Stock (Chem. Service)

7.2.15 Azobenzene Stock (Chem. Service)

7.2.16 1-Methyl Naphthalene Stock (Chem. Service)

7.2.17 (DFTPP) Decafluorotriphenylphosphine (protocol)

NOTE: Individual compound stock standards prepared from NEAT. (Table 12)

7.3 Internal Standard Mix — S.V. CLP Internal Standard Mix (Absolute) (See Table 11).

7.3.1 Consisting of 1,4-Dichlorobenzene-d_4, Naphthalene-d_8, Acenaphthene-d_{10}, Phenanthrene-d_{10}, Chrysene-d_{12}, Perylene-d_{12}.

7.3.2 Stored at -10°C to -20°C in amber glass vials with Teflon lined caps.

7.3.3 Internal standard diluted to a concentration of 1000ug/ml in methylene chloride is added to all samples and at 20uL per 1ml of sample, 10uL per 0.5mL of sample extract to give a concentration of 40ng on column for each sample.

7.4 Ultra high purity helium used as a carrier gas to all instruments

8.0 PROCEDURE

8.1 Daily procedure

8.1.1 Check gas cylinders in the beginning of the day to ensure proper pressure and adequate abundance of gas

8.1.1.1 Change gas cylinder if amount falls below 500psi.

- Check each instrument assigned to cylinder to be changed and make sure all of the GC ovens are at ambient temp (40-50°C), and no instruments are in the middle of analysis.
- Place full cylinder next to empty one in as close proximity to where it will stay as possible.
- Make sure empty cylinder being removed has the valve completely shut off.
- With large crescent wrench unscrew regulator from cylinder as quickly as possible and immediately place on full cylinder, tighten and turn valve on. Open valve as far as it will go and back off ¼ turn to prevent valve sticking. This procedure should be completed as quickly as absolutely possible to minimize ambient air entering the gas lines and to avoid total loss of gas pressure to the instruments.
- Check fittings on regulator with snoop (soapy water) for air tightness. If there is a leak at fitting the snoop will bubble, if this occurs, check fitting and tighten or replace as necessary.

8.1.1.2 Log in cylinder pressure (PSI) on gas monitoring sheet.

8.1.2 Check refrigerator/freezer unit where samples and standards are stored.

8.1.2.1 Using an NIST Calibrated Mercury Thermometer check that the temperatures are being held at 1°C to 4.4°C for the refrigerator and -10°C to -20°C for the freezer.

8.1.2.2 Log temperature for each in the Zone Calibration Check Refrigeration Log Book to the nearest 0.5°C.

8.1.3 Set daily working standards to be used for the day out of freezer to warm to room temperature before analysis.

8.1.4 Rinse and refill solvent vials used for sample preparation and dilutions. Empty waste vials. These vials are kept at sample prep tables.

8.2 Analytical Procedure

8.2.1 Check instrumentation for any obvious problems (i.e., broken syringe, power failure).

8.2.2 If samples analyzed the night before were dirty, set injection port and GC oven to cool to do daily maintenance. Turn off injection port. Set oven to 30°C.

8.2.3 Briefly review the previous day's analyses by processing the samples in accordance with the "*Enviroquant GC/MS Data Processing*" SWL-OS-401, check for any major system problems (i.e., broken column, air leak, blown filament, etc.) and check to confirm that the previous sequence has been completed. If problems exist, inform supervisor and consult for corrective action. If you are operating two instruments, continue operating the functional instrument.

8.2.4 Perform daily maintenance on instrument injection port (Attachment 10) and column. Care must be taken to not touch any parts with bare hands unless absolutely unavoidable. Use tweezers for placement of new parts.

8.2.4.1 Before opening the closed injection port system make note of carrier gas pressure on the HP5890GC/HP5970 MSD and the electronic pressure controller (EPC) on HP5890/HP5971/HP5972 MSD. On the HP5971/72 make note of pressure at a given temp (i.e., 50°C → 7.7 pressure) and then set pressure to zero.

8.2.4.2 Change septa. Remove septum retainer nut and change out septa using tweezers. Save old septa to plug disconnected column; replace septum retainer nut finger tight. Do not over tighten, over tightening puts too much pressure on septum and allows syringe needle to push out chunks of the septum clogging the injection port liner and causing a leak.

8.2.4.3 Loosen insert weldment assembly and remove injection port liner and o-ring.

8.2.4.4 Disconnect column from injection port. Remove column nut and old ferrule from column. Use old septum to plug off column, to avoid excessive air being pulled through the system.

8.2.4.5 Remove insulator cup and reducing nut from injection port. Discard used seal and washer from inside reducing nut. Replace with cleaned or new seal and washer. Use tweezers to handle seals and washers.

Do no touch with hands. Rinse new seal and washer with methylene chloride before placing back in reducing nut. Double check to make sure washer is on the bottom of the reducing nut with the seal, tip down, on top of washer. (Tip on seal should be down towards small end of reducing nut). Replace reducing nut, tightening completely, and replace insulating cup. Insulation inside insulating cup should be intact and relatively snug around reducing nut.

8.2.4.6 Install new injection port liner into top of injection port, using tweezers or forceps. Put a new o-ring around top of injection port liner. This can easily be done using two tweezers and pulling o-ring around liner. Make sure o-ring is as flat as possible against the injection port shell weldment. Tighten insert weldment assembly back down onto shell weldment. Make sure insert weldment is properly aligned with the tab in the back down in the large hole on shell weldment and straight over the threads. Tighten completely.

8.2.4.7 Re-install column to injection port. Place column nut back on column and put tapered .4mm ID Graphite/Vespel Ferrule on column, with wide end of Ferrule fitting into column nut. Clip column. If dirty samples were analyzed, 8-10 inches should be clipped off column. If clean samples were analyzed, clip 4-8 inches off. Extreme care must be taken while clipping a column to ensure a flat, even cut on end of column. Set column 4-5 mm above top of Ferrule and mark position under column nut with white out. Screw column nut back up into reducing nut on injection port, being careful to reset column according to white out mark to ensure proper column height within injection port liner.

8.2.4.8 Gently tighten detector interface nut on detector end of column to prevent air leak at this point. Do not over-tighten.

8.2.4.9 Once all maintenance is completed and returned to a closed system, check carrier gas pressure on dial on HP5890/HP5970 (to make sure it has returned to the same pressure as before daily maintenance), to ensure no major obvious air leaks are occurring, and reset EPC pressure on HP5890/HP5971/72 (i.e., 50°C oven temp = 7.7 pressure). If EPC pressure does not stabilize at set point within a few seconds there is probably a leak at some point where maintenance was just completed.

8.2.4.10 Turn injection port temperature back on. Turn oven temperature to 150°C and let bake for 10 to 15 minutes. This will facilitate ridding the instrument of any air/water within the system from the column being open.

8.2.5 Prepare an aliquot of DFTPP performance check solution (Table 13) and continuing calibration standard (SSTD050, Table11) while instrument is baking out. Also during this time check disk space on the chem station and the

Enviroquant system to ensure adequate disk space to acquire data for a twelve hour time period for all instruments on the data system.

8.2.5.1 In a 1.8mL clear glass crimp top vial with a 250uL glass insert add approximately 30uL of 50ng DFTPP solution. 30uL should be sufficient for several injections if needed. Do the same with the current continuing calibration solution. Label vials with solution name, solution lab ID number, (i.e., DFTPP 1-208-12; SSTD050 1-213-12).

8.2.5.2 Check disk space on Enviroquant/ChemStation system as follows on Windows 95/98 systems:

- Start/Programs/Windows Explorer
- Click on "C:" drive
- Click on "Hpchem," choose instrument number, click on "Data"
- Delete all data except for the most recent week

8.2.6 Inject 2uL of the 50ng DFTPP solution.

8.2.7 While DFTPP is running, begin reviewing previous day's analysis that was previously processed in Section 8.2.3. (See SOP SWL-OS-303 "*Semivolatile Data Review*"). Check each sample analyzed for the following:

8.2.7.1 Internal standards

8.2.7.2 Surrogate/Spike recoveries (See Table 2 for surrogate and QC-Blue-Book for up to data spike recoveries.)

8.2.7.3 Linearity and saturated peaks

8.2.7.4 All samples within 12 hour clock

8.2.7.5 Major analytical problems with samples (i.e., run terminated early; air leaks).

Note: Each of the above can be checked by clicking on "SWOK" and view reports on screen"

8.2.7.6 Remedial action if criteria are not met

- Waters: If peaks are saturated, the sample must be diluted to get the highest-level target compounds within linear range and reanalyzed.
- Soils: If peaks are saturated, the sample must be diluted to get the highest-level target compounds within linear range. If a dilution greater than 30 fold is required, then sample may need to be re-extracted as a medium level extract and reanalyzed. Consult

supervisor for instructions. (IS intensities should be at least 10% or greater in the sample for correct dilution.)

- If any target analyte has a concentration above 1000ng on column, then following sample must be checked for carry-over. (See SOP SWL-OS-303 "*Semivolatile Data Review*").

- If sample is analyzed at medium level, laboratory control spike and blank analyses must also be performed for that level.

- If surrogate recovery for semivolatiles in any sample is less than 10%, the sample must be re-extracted and reanalyzed. If two or more acid surrogates or two or more base/neutral surrogates are outside of QC limits in any sample not including the advisory surrogates 1,2-Dichlorobenzene-d4 or 2-Chlorophenol-d4 the sample must be re-extracted and re-analyzed. If one acid surrogate and/or one base surrogate is outside of QC limits, and the recovery is greater than 10%, the data must be flagged and noted in client's narrative.

  NOTE: Clients QAP or QAPP must be followed, which in some cases does not allow for any surrogates outside QC limits without re-extracting.

  No surrogates can be out of QC limits for extraction blanks (not including advisory surrogates: see section 8.2.7.6.) If so, all samples extracted with blanks that have surrogates out must be re-extracted. Both sets of data will be reported if the re-extracts are not completed within the required holding times (consult the section supervisor).

- Extraction Blank Contamination: Extraction Blanks cannot have target analytes of interest above the PQL. The only exceptions could be the common laboratory contaminates of phthalate, which could not be detected above 5 times the PQL. Anytime phthalate is detected above the PQL the blank must be reanalyzed and verified by supervisor as usable.

  NOTE: Clients QAP or QAPP must be followed, which in some cases does not allow for any analytes to be above reporting levels.

  When any other target analytes in blank are above PQL, all associated samples containing these analytes must be re-extracted. See supervisor for directions on any contaminated blanks.

  NOTE: All Blank contamination must be reported.

- All samples that are re-extracted must be logged-in on the "Re-extract Tracking Sheet" (Attachment 1), and turned into the Supervisor.

- If re-extraction is required for a sample, fill out a Non-Conformance Report (GA-105-NCR-F Attachment 3). Include a copy of the extraction sheet and a copy of spike report or tabular report documenting why sample is being re-extracted. Non-Conformance Report must be signed by Organic Program Manager/Supervisor/Group Leader prior to being submitted to extractions department.

8.2.8 When the DFTPP run is complete, check to see if the tuning criteria has been met (see Table 1), and that all peaks in DFTPP solution are accounted for and less than 20% 4,4-DDT breakdown is present (see Formula 9.6), or poor recovery of pentachlorophenol, or tailing of benzidine (Attachment 7, 8, and 9).

NOTE: Benzidine and pentachlorophenol should be present at their normal responses, and no peak tailing should be visible. If so, start the standard analysis, and print the DFTPP report. Note in the Run Log Comment section that DFTPP passed. If DFTPP does not pass, re-shoot DFTPP or tune the mass spec first, then shoot DFTPP. If the tune does not pass after 3 attempts, consult your supervisor for instruction.

8.2.9 Inject 2uL of the continuing calibration, or if an initial calibration is being performed, set up a sequence for standards in the calibration. Start the sequence running the continuing calibration and prep and load the remaining standards.

8.2.10 If the standard is a continuing calibration, not an initial calibration running, set out 3-5 samples (either samples requiring re-analysis, or samples being initially run) to warm to room temperature. Add internal standards to samples if necessary once all have warmed. Prepare an aliquot of each sample for injection.

NOTE: All vials for sample analysis must be labeled with entire lab id number (i.e., 29859.02, BL0618WA-29859).

8.2.10.1 Internal standards solution is added to each sample at 40ng/uL on column. Add 20uL of the 1000ng/uL solution to each 1.0mL extract and 10uL for each 0.5mL extract of sample to achieve the appropriate 40ng/2uL concentrations.

8.2.11 When the continuing calibration has finished, check to see if the acceptance criteria has been met (Table 8 & 9) and that the data system has correctly identified all isomeric compounds (Tables 5 & 6). If so, load the 3-5 samples, and start the sequence. If the standard does not pass, and if no major problems exist (e.g., very low RFs for SPCCs <0.05, % Deviation> 20 for CCC etc.), rerun the continuing calibration. If the second calibration does not pass, set up an initial calibration sequence. All standards should be reviewed immediately after analysis to make sure it passes before samples are analyzed. Submit for secondary review after checking standard.

8.2.12 While the short sequence of samples (or initial calibration) is being analyzed, continue reviewing previously processed data.

8.2.13 Print all samples together after they've been edited. Each sample is to be assembled in the following order

8.2.13.1 Sample Report (Form 1 type report)

8.2.13.2 Surrogate/Spike Report

8.2.13.3 Internal Standard Report

8.2.13.4 Total Ion Chromatogram

8.2.13.5 Target Quant. Report

8.2.13.6 Manual Integration(s)

8.2.13.7 Target Compound Spectra

8.2.13.8 TIC Alkane Report (As required)

8.2.13.9 TIC Spectra (As required)

8.2.14 Print a run log for each 12-hour clock, in accordance with SOP SWL-OS-302, "Semivolatile Run Logbook Maintenance."

NOTE: Highlight runs for each episode with yellow highlighter.

8.2.14.1 Double check all samples are within 12-hour clock on run log.

8.2.14.2 If there is a problem with any runs, note in right margin what it is (ex. 2 internal stds low, 2 surrogates low, rerun @2x, re-extracting).

8.2.14.3 Check Client ID, Lab ID, weights/vol., dilutions.

8.2.14.4 Double check that correct method and sublist was used.

8.2.15 Upon completion of data review, determine the remaining samples to be analyzed. (Re-analysis or dilutions from reviewed data or new samples.)

8.2.15.1 Set samples out to warm to room temperature.

8.2.15.2 When samples have warmed to room temperature, add internal standards, if not previously added, or do dilutions as necessary.

8.2.16 Add additional samples to be analyzed to short sequence already running.

8.2.17 After entire sequence has been typed in, pause the sequence between runs and save the sequence on the chem station. Print a hard copy of the sequence to check for correctness and proper vial placement. Restart sequence where it was paused. Keep hard copy of sequence with Run LogBook.

8.2.18 For any corrections or errors made cross out error with a single line and initial and date.

8.2.19 After each sample has been printed and put together as in Section 8.2.14 (but entire episode is not complete), keep each sample for an episode together with the run log for that clock.

8.2.19.1 Attach a note to each group of data stating why it is not complete and what is remaining on it (ex. Rerun 29864.02 @ 10X, 29864.01 sent for re-extract. 29864.06 - .12 running now, etc.)

8.2.19.2 Keep sample extraction sheet with data.

8.2.20 Completed episodes must be assembled and turned in for secondary review in the following order:

8.2.20.1 Extraction Sheet (Attachment 4).

8.2.20.2 Copies of Run Log Book pages pertaining to the episode for all data analyzed (for each page with QC, samples, calibration, initial calibrations).

8.2.20.3 Data Review Sheet (see Attachment 2)

8.2.20.4 Back-up Blank Sheet (If needed) (See Attachment 12 and SOP SWL-GA-133 "*Usage of Blanks and Backup Blanks*")

8.2.20.5 Sample Data

- SBLK
- LCS/LCSD
- Samples

8.2.20.6 Re-extracts (if needed) arranged in same order as original data.

8.2.20.7 Extra Data

8.2.21 As time permits, data should be worked up as it is running. Data ran overnight or during another shift should be processed and reviewed the following shift as stated beginning in Section 8.2.3.

9.0 CALCULATIONS

9.1 Water

$$\text{Concentration (ug/L)} = \frac{(Ax)\ (Is)\ (Vt)}{(Ais)\ (RF)\ (Vo)\ (Vi)}$$

Where:

Ax	=	Area of the characteristic ion for the compound to be measured.
Ais	=	Area of the characteristic ion for the internal standard.
Is	=	Amount of internal standard injected in nanograms (ng).
Vo	=	Volume of water extracted in mililiters (mL).
Vi	=	Volume of extract injected (uL).
Vt	=	Volume of total extract (uL).
RF	=	Average Response Factor

9.2 Sediment/Soil

$$\text{Concentration (ug/Kg)} = \frac{(Ax)\ (Is)\ (Vt)}{(Ais)\ (RF)\ (Ws)\ (Vi)\ (D)}$$

(Dry weight basis)

Where:

Ax, Is, Ais = Same as given for water, above.
Vt = Final volume of low level total extract (uL).
Vi = Volume of extract injected (uL).
D = $\frac{100 - \%\ \text{moisture}}{100}$
Ws = Weight of sample extracted (grams).
RF = Average Response Factor

9.3 An estimated concentration for Non-TCL components tentatively identified shall be quantified by the internal standard method. For quantification, the nearest internal standard shall be used.

9.4 Internal Standard added to all samples

$$\frac{\text{1000ng/uL Internal Standard Solution x 20uL}}{\text{1000uL Sample}} = \text{40ng/2uL per sample}$$

9.5 Common Sample Dilutions

- 2x = 500uL sample + 500uL CH_2Cl_2 + 10uL Int.Std.
- 5x = 200uL sample + 800uL CH_2Cl_2 + 16uL Int.Std.
- 10x = 100uL sample + 900uL CH_2Cl_2 + 18uL Int.Std.
- 20x = 50uL sample + 950uL CH_2Cl_2 + 19uL Int.Std.
- 50x = 20uL sample + 980uL CH_2Cl_2 + 20uL Int.Std.
- 100x = 10uL sample + 990uL CH_2Cl_2 + 20uL Int.Std.

9.6 % Breakdown for DDT

$$\%\ BD = \frac{DDE_{(PA/PH)} + DDD_{(PA/PH)}}{DDT_{(PA/PH)} + DDE_{(PA/PH)} + DDD_{(PA/PH)}} \times 100$$

BD = Breakdown
(PA/PH) = Peak area or peak height of analyte.
NOTE = % breakdown of DDT in DFTPP must be less than 20%.

9.7 See Table 14 for remaining calculations. for linearity, surrogate recovery, matrix spike and lab spike recovery, and percent RSDs.

10.0 QUALITY ASSURANCE/QUALITY CONTROL

10.1 Extraction Blanks and Laboratory Control Spikes are extracted at a minimum of 5% of samples being extracted.

10.2 Any sample or QC sample with a surrogate less than 10% must be re-extracted and re-analyzed. If two or more acid surrogates or two or more base/neutral surrogates are outside QC limits in any sample (not including advisory surrogates; see 8.2.7.6), the sample must be re-extracted and re-analyzed. If one acid and/or one base surrogate is outside of QC limits and the recovery is greater than 10%, the data must be flagged and noted in clients narrative.

NOTE: Clients QAP or QAPP must be followed, which in some cases does not allow for any surrogates outside QC limits without re-extracting.

10.3 No surrogates can be outside QC limits for extraction blanks (not including advisory surrogates; see 8.2.7.6). If the extraction blank has surrogates outside QC limits, all samples extracted with that blank must be re-extracted.

10.4 Extraction blanks cannot have target analytes above the PQL. The only exceptions could be the common laboratory contaminate of phthalate, which could not be detected above 5 times the PQL. Anytime phthalate is detected above the PQL the blank must be re-analyzed and verified by supervisor as "OK" to use.

NOTE: Clients QAP on QAPP must be followed, which in some cases does not allow for any analytes to be above reporting levels. When target analytes in the extraction blank and laboratory control spikes (other than spike compounds) are above the PQL all associated samples containing these analytes must be re-extracted. Consult supervisor for instructions on any contaminated QC.

10.5 Laboratory Control Spike

10.5.1 The Laboratory Control Spike (LCS and LCSD) are internal laboratory QC samples designed to show the capability of our laboratory to be in compliance with the protocol.

10.5.2 An acceptable LCS and LCSD should meet the internal standard criteria stated in SWL-OS-303 "*Semivolatile Data Review*" and the surrogate recoveries in Table 2. The percent recovery for each of the compounds in the LCS and LCSD should be within recovery limits shown in the QC Method Reference "Blue Book". Limits are derived using a control chart program as per the "*Control Charts*" SOP (SWL-GA-112).

10.5.3 If the compounds are outside QC limits, corrective action is needed immediately, beginning with rerunning the LCS to verify that it was injected correctly.

10.6 Matrix Spikes — MS/MSDs will be analyzed at a rate of 1 MS/MSD per batch of 20 or fewer samples. A minimum of the 11 compounds will be used; however all compounds can be spiked.

10.7 Each analytical instrument is calibrated daily with a DFTPP tuning standard (see Table 1) and a calibration standard with all compounds of interest.

10.7.1 Tune

10.7.1.1 Before analysis of samples can begin, the instrument must meet all calibration criteria. The first step in the calibration of a Semi-volatile GC/MS system is to demonstrate the ionization and fragmentation of standard mass spectral tuning compounds. This, as well as a sensitivity check, is accomplished with the use of Decafluorotriphenylphosphine (DFTPP), an EPA specified compound. This standard is run daily to validate the GC/MS system and to begin the 12-hour clock. DFTPP is analyzed by direct injection of a 50ng aliquot into the instrument.

10.7.1.2 When the analysis is complete, the tune is checked against standard criteria (See Table 1 for Tune Criteria). SW8270B tune criteria for DFTPP must be met before any other calibration standards or sample analysis is performed.

10.7.1.3 If the tune fails to meet the criteria, DFTPP is rerun. If it fails again, the instrument must be re-tuned through "Auto Tune" or "Manual Tune", then DFTPP should be run again. If all criteria are met, the calibration sequence is run, but if it fails again, a supervisor is contacted because instrument maintenance such as source cleaning may be required.

10.7.2 Initial Calibration

10.7.2.1 Once DFTPP has met its tune criteria, calibration may begin. Initial calibration of the GC/MS is established and validated by the analysis of EPA traceable standards at five concentration levels. Standard levels are 20, 50, 80, 120 and 160uL, but other levels may be used. These standards are made up in methylene chloride before analysis by GC/MS. The internal standard calibration procedure is used. In addition to surrogate recovery compounds, standards and sample extracts are spiked with internal standards that span the retention time range of the analytes of interest.

10.7.2.2 The Response Factors (RF) for the analytes in the standard are calculated and stored in a Enviroquant Method File. Using the RFs from the initial calibration, the percent relative standard deviation (%RSD) for all compounds is calculated utilizing the formula shown in Table 14. The criteria the initial calibration must meet to be acceptable is found in Table 8 and 9 for CCC's and SPCC's.

10.7.2.3 If the five-point does not meet criteria each standard is checked for the need of a possible rerun, in case the standards had a bad injection. If re-analyzing a standard will not solve the problem, a supervisor is contacted.

10.7.2.4 If the initial calibration does meet the criteria stated in Table 8 and 9, and there is time remaining in the 12-hour time period, samples may be run.

- If the % RSD of any compound is 15% or less, then the relative response factor is assumed to be constant over the calibration range, and the average relative response factor may be used for Quantitation.
- If the % RSD of any compound is greater than 15%. The compounds should be checked to verify that all integration's were performed properly, that each calibration level appears to be at the correct concentration levels and that the system is operating properly (i.e. normal response factors and retention times). If all of this is in order, then a calibration curve must be constructed of area ratios (A/Ais) versus concentration using first or higher order regression fit of the five calibration points (i.e., linear regression). The use of calibration curves is a recommended alternative to average response factor calibration and a useful diagnostic of standard preparation accuracy and absorption activity in the chromatographic system.
- If Linear Regression does not work (<0.99), the system will be re-calibrated, if compound detected in samples.

10.7.3 Calibration Verification

10.7.3.1 A calibration verification of the GC/MS uses the same EPA traceable standards, but at only one level: 80ng/uL. The analysis is performed in the same manner as stated before in the initial calibration. The calibration verification checks the same compounds as the initial calibration, and the criteria the calibration verification must meet to be acceptable are also found in Table 8 and 9.

All other compounds should have a %Drift less than 30%.

10.7.3.2 The calibration verification is performed after the DFTPP and must meet all criteria before any other analysis continues. If the CCC/SPCC criteria are not met, the standard must be rerun, as it is possible, the standard was not prepared correctly or that there was a bad injection. However, if the second standard still doesn't meet CCC/SPCC criteria, and initial calibration must be run or corrective action performed.

10.7.3.3 If the calibration verification does meet the criteria stated in Tables 8 and 9, samples may be run. Samples are quantitated based on the criteria stated above in Section 10.7.2.4.

11.0 WASTE DISPOSAL/POLLUTION PREVENTION

11.1 All waste solvent is collected in a designated area and disposed of by a hazardous waste department designee, in accordance with SOP SWL-GA-114 "*Hazardous Waste Management Plan.*"

11.2 All sample vials are collected in designated boxes at each instrument, and periodically picked up and disposed of by hazardous waste designee and in accordance with SOP SWL-GA-114 "*Hazardous Waste Management Plan.*"

12.0 METHOD PERFORMANCE

12.1 Surrogate Recovery Limits (Table 2)

12.2 Matrix Spike Recovery Limits (Table 3)

12.3 Laboratory Control Spike Limits (QC Blue Book)

12.4 DFTPP – Tuning Criteria (Table 1)

12.5 Continuing Calibration SPCC/CCC Criteria (Tables 8 & 9)

12.5.1 SPCC – Semivolatile System Performance Check Compounds

12.5.2 CCC – Semivolatile Calibration Check Compounds

12.6 MDL run annually and must be below reporting limits (PQL's). All MDLs stored with section supervisor and QC Red Book.

13.0 REFERENCES

13.1 SW846-8270C, Revision 3, December 1997

13.2 Hewlett Packard, chemical analysis columns and supplies catalog 1996-97.

13.3 "*Semivolatile GC/MS Data Processing*" SOP SWL-OS-401.

13.4 "*Semivolatile Data Review*" SOP SWL-OS-303.

13.5 "*Semivolatile Run Logbook Maintenance*" SOP SWL-OS-302.

13.6 "*Hazardous Waste Management Plan*", SWL-GA-114.

13.7 "*Laboratory Safety Plan*", SWL-GA-111.

13.8 "*Guide to Semivolatile GC/MS Standards Log*", SWL-OS-309.

14.0 DEFINITIONS

14.1 Terminology

14.1.1 Standard Operating Procedure (SOP): Written narratives that describe step-by-step instructions and procedures specific to the laboratory.

14.1.2 Batch: ..... Daily analytical sequence consisting of the daily tune, calibration, method blank, QC spikes, and samples analyzed within a 12-hour time clock.

14.1.3 Extra Data: ..... Data classified as non-reportable, but stored by the laboratory with the pertinent episode information for reference purposes.

14.1.4 Extraction Sheet: ..... Information generated in Extraction. Sheet is specific to each set of samples and contains, at a minimum, the Client, Client ID, SWLO ID, pH, %Moisture, and Extraction Information.

14.1.5 Lab Prep Batch: ..... Identification assigned when the Method Blank is analyzed and used by all samples corresponding to that Method Blank.

14.1.6 Standard Tabular Report: ..... Type of data package report generated by the Reporting Department from information.

14.1.7 Sequence: ..... Same as Batch

14.1.8 Test Code: ..... Code assigned to each sample to determine which department sample is for and what method sample is to be analyzed by.

14.2 Acronyms

14.2.1 RT ..... Retention Times

14.2.2 MS/MSD ..... Matrix Spike/Matrix Spike Duplicate

14.2.3 TIC ..... Tentatively Identified Compound

14.2.4 MSDS ..... Material Safety Data Sheets

14.2.5 GC ..... Gas Chromatogram

14.2.6 GC/MS ..... Gas Chromatography/Mass Spectroscopy

14.2.7 HP ..... Hewlett Packard

14.2.8 NIST ..... National Institute of Standards and Technology

14.2.9 DFTPP ..... Decafluorotriphenylphosphine

14.2.10 PQL..... Practical Quantitation Limit

14.2.11 IS..... Internal Standards

14.2.12 CCC ..... Calibration Check Compound

14.2.13 SPCC..... System Performance Check Compound

14.2.14 LCS/LCSD.... Laboratory Control Spike/Laboratory Control Spike Duplicate

15.0 TABLES — See following pages.

16.0 ATTACHMENTS

16.1 ATTACHMENT 1: Re-Extract Tracking Sheet

16.2 ATTACHMENT 2: Data Review Sheet

16.3 ATTACHMENT 3: Non-Conformance Report

16.4 ATTACHMENT 4: Sample Analysis/Extraction Log

16.5 ATTACHMENT 5: Extract Receipt Log

16.6 ATTACHMENT 6: Semivolatiles GC/MS Run Log

16.7 ATTACHMENT 7: DFTPP (Page 1 of 3)

16.8 ATTACHMENT 8: DFTPP (Page 2 of 3)

16.9 ATTACHMENT 9: DFTPP (Page 3 of 3)

16.10 ATTACHMENT 10: Split/Splitless (Multimode) Inlet

16.11 ATTACHMENT 11: Enviroquant Window

16.12 ATTACHMENT 12: Back-up Blank Usage Form

Table 1
DFTPP Key Ions and Abundance Criteria

Mass	Ion Abundance Criteria			
51	30% to	60%	of mass	198
68	Less than	2%	of mass	69
70	Less than	2%	of mass	69
127	40% to	60%	of mass	198
197	Less than	1%	of mass	198
198	Base peak	100%	relative abundance	
199	5% to	9%	of mass	198
275	10% to	30%	of mass	198
365	Greater than	1%	of mass	198
441	Present but less than mass			443
442	Greater than	40%	of mass	198
443	17% to	23%	of mass	442

Table 2
Surrogate Spike Recovery Limits

Fraction	Surrogate Compound	Water	Low/Medium Soil
BN	Nitrobenzene-d5	35 - 114	23 - 120
BN	2-Fluorobiphenyl	43 - 116	30 - 115
BN	p-Terphenyl-d14	33 - 141	18 - 137
Acid	Phenol-d5	10 - 94	24 - 113
Acid	2-Fluorophenol	21 - 100	25 - 121
Acid	2,4,6-Tribromophenol	10 - 123	19 - 122
Advisory Surrogates			
Acid	2-Chlorophenol-d4	33 - 110	20 - 130
BN	1,2-Dichlorobenzene-d4	16 - 110	20 - 130

* Method Limits

Table 3
Matrix Spike Recovery Limits

Fraction	Matrix Spike Compound	Water	RPD	Sediment/Soil	RPD
BN	1,2,4-Trichlorobenzene	39 - 98	28	38 - 107	23
BN	Acenaphthene	46 - 118	31	31 - 137	19
BN	2,4-Dinitrotoluene	24 - 96	38	28 - 89	47
BN	Pyrene	26 - 127	31	35 - 142	36
BN	N-Nitroso-Di-n-Propylamine	41 - 116	38	41 - 126	38
BN	1,4-Dichlorobenzene	36 - 97	28	28 - 104	27
Acid	Pentachlorophenol	9 - 103	50	17 - 109	47
Acid	Phenol	12 - 89	42	26 - 90	35
Acid	2-Chlorophenol	27 - 123	40	25 - 102	50
Acid	4-Chloro-3-Methylphenol	23 - 97	42	26 - 103	33
Acid	4-Nitrophenol	10 - 80	50	11 - 114	50

* Recovery Limits from EPA/CLP are advisory.

Table 4
Characteristic Ions for Pesticide/PCBs

Parameter	Primary Ion	Secondary Ion(s)
BHC	183	181, 109
Beta-BHC	181	183, 109
Delta-BHC	183	181, 109
Gamma-BHC (Lindane)	183	181, 109
Heptachlor	100	272, 274
Aldrin	66	263, 220
Heptachlor Epoxide	353	355, 351
Endosulfan I	195	339, 341
Dieldrin	79	263, 279
4,4'-DDE	246	248, 176
Endrin	263	82, 81
Endosulfan II	337	339, 341
4,4'-DDD	235	237, 165
Methoxychlor	227	228
Chlordane (alpha and/or gamma)	373	375, 377
Toxaphene	159	231, 233
Aroclor-1016	222	260, 292
Aroclor-1221	190	222, 260
Aroclor-1232	190	222, 260
Aroclor-1242	222	256, 292
Aroclor-1248	292	362, 326
Aroclor-1254	292	362, 326
Aroclor-1260	360	362, 394
Endrin Ketone	317	67, 319

Table 5A
Characteristic Ions for Semivolatile TCL Compounds

Parameter	Primary Ion	Secondary Ion(s)	Parameter	Primary Ion	Secondary Ion(s)
Phenol	94	65,66	2,4-Dinitrophenol	184	63,154
bis(2-Chloroethyl) Ether	93	63,95	4-Nitrophenol	109	139,65
2-Chlorophenol	128	64,130	Dibenzofuran	168	139
1,3-dichlorobenzene	146	148,113	2,4-Dinitrotoluene	165	63,182
1,4-Dichlorobenzene	146	148,113	2,6-Dinitrotoluene	165	89,121
Benzyl Alcohol	108	79,77	Diethylphthalate	149	177,150
1,2-Dichlorobenzene	146	148,113	4-Chlorophenyl-phenylether	204	206,141
2-Methylphenol	108	107	Fluorene	166	165,167
bis(2-chloroisopropyl) Ether	45	77,79	4-Nitroaniline	138	92,108
4-Methylphenol	108	107	4,6-Dinitro-2-Methylphenol	198	182,77
N-Nitroso-Di-Propylamine	70	42,101,130	N-Nitrosodiphenylamine	169	168,167
Hexachloroethane	117	201,199	4-Bromophenyl-phenylether	248	250,141
Nitrobenzene	77	123,65	Hexachlorobenzene	284	142,249
Isophorone	82	95,138	Pentachlorophenol	266	264,268
2-Nitrophenol	139	65,109	Phenanthrene	178	179,176
2,4-Dimethylphenol	107	121,122	Anthracene	178	179,176
Benzoic Acid	122	105,77	Di-N-Butylphthalate	149	150,104
bis(-2-Chloroethoxy) Methane	93	95,123	Fluoranthene	202	101,100
2,4-Dichlorophenol	162	164,98	Pyrene	202	101,100
1,2,4-Trichlorobenzene	180	182,145	Butylbenzylphthalate	149	167,279
Naphthalene	128	129,127	3,3-Dichlorobenzidine	252	253,125
4-Chloroaniline	127	129	Benzo (a) Anthracene	228	229,226
Hexachlorobutadiene	237	235,272	bis(2-Ethylhexyl)Pthalate	149	167,279
2,4,6-Trichlorophenol	196	198,200	Chrysene	228	226,229
2,4,5-Trichlorophenol	196	198,200	Di-N-Octyl Phthalate	149	———
2-Chloronaphthalene	162	164,127	Benzo (b) Fluoranthene	252	253,125
2-Nitroaniline	65	92,138	Benzo (k) Fluoranthene	252	253,125
Dimethyl Phthalate	63	194,164	Benzo (a) Pyrene	252	253,125
Acenaphthylene	152	151,153	Indeno (1,2,3-cd) Pyrene	276	138,227
3-Nitroaniline	138	108,92	Dibenz (a,h) Anthracene	78	139,279
Acenaphthene	153	152,154	Benzo (g,h,i) Perviene	76	138,277
Carbazole	167	166, 139			

Table 5B
Characteristic Ions for Non-TCL Semivolatile Compounds

Parameter	Primary Ion(s)	Secondary Ion(s)	Parameter	Primary Ion(s)	Secondary Ion(s)
2-Picoline	93	66, 92	2,3,4,6-Tetrachlorophenol	232	232, 131, 230, 166, 234, 168
Aniline	93	66, 65	1,3,5-Trinitrobenzene	75	75, 74, 213, 120, 91, 63
N-Nitrosomethylethylamine	88	42, 88, 43, 56	Benzidine	184	92, 185
Thiophenol (Benzenethiol)	110	110, 66, 109, 84	Phorate	75	75, 121, 97, 93, 260
Methyl Methanesulfonate	80	80, 79, 65, 95	Sulfallate	188	188, 88, 72, 60, 44
Ethyl methanesulfonate	79	79, 109, 97, 45, 65	Phenacétin	108	180, 179, 109, 137, 80
Hexachloropropene	213	213, 211, 215, 117, 106, 141	Dimethoate	87	87, 93, 125, 143, 229
N-Nitrosopyrrolidine	100	100, 41, 42, 68, 69	4-Aminobiphenyl	169	169, 168, 170, 115
Acetophenone	105	71, 105, 51, 120	α,α-Dimethylphenylamine	58	58, 91, 65, 134 42
o-Toluidine	106	106, 107, 77, 51, 79	Pronamide	173	173, 175, 145, 109, 147
3-Methylphenol	107	107, 108, 77, 79, 90	Disulfoton	88	88, 97, 89, 142, 186
N-Nitrosopiperidine	114	42, 114, 55, 56, 41	Methyl parathion	109	109, 125, 263, 79, 93
1,4-Phenylenediamine	108	108, 80, 53, 54, 52	Dimethylaminoazobenzene	225	225, 120, 77, 105, 148, 42
1-Chloronaphthalene	162	127, 164	Kepone	272	272, 274, 237, 178, 143, 270
5-Chloro-2-methylaniline	106	106, 141, 140, 77, 89	Parathion	109	109, 97, 291, 139, 155
Isosafrole	162	162, 131, 104, 77, 51	3,3'-Dimethylbenzidine	212	212, 106, 196, 180
2-Naphthylamine	143	115, 116	Methapyrilene	97	97, 50, 191, 71
1,4-Naphthoquinone	158	158, 104, 102, 76, 50, 130	Isodrin	193	193, 66, 195, 263, 265, 147
Safrole	162	162, 104, 77, 103, 135	Aramite	185	185, 191, 319, 334, 197, 321
Diphenylamine	169	168, 167	Chlorobenzilate	251	251, 139, 253, 111, 141
1,2,4,5-Tetrachlorobenzene	216	216, 214, 179, 108, 143, 218	Famphur	218	218, 125, 93, 109, 217
1-Naphthylamine	143	143, 115, 89, 63	7,12-Dimethylbenz(a)anthracene	256	256, 241, 239, 120
5-Nitro-o-toluidine	152	77, 152, 79, 106, 94	2-Acetylaminofluorene	181	181, 180, 223, 152
1,3-Dinitrobenzene	168	168, 76, 50, 75, 92, 122	3-Methylcholanthrene	268	268, 252, 253, 126, 134, 113
Diallate (cis or trans)	86	86, 234, 43, 70	Dibenz (a,j)acridine	279	279, 280, 277, 250
Diallate (trans or cis)	86	86, 234, 43, 70	1,2:4,5-Dibenzopyrene	302	302, 151, 150, 300
Pentachloronitrobenzene	237	237, 142 214, 249, 295, 265	Hexachlorophene	196	196, 198, 209, 211, 406, 408
4-Nitroquinoline-1-oxide	174	174,101, 128, 75, 116			

Table 6
Characteristic Ions for Surrogate and Internal Standards for Semivolatile Organic Compounds

Surrogate Standards			Internal Standards		
Parameter	Primary Ion	Secondary Ion(s)	Parameter	Primary Ion	Secondary Ion(s)
Phenol-d5	99	42, 71	1,4-Dichlorobenzene-d4	152	115
2-Fluorophenol	112	64	Naphthalene-d8	136	68
2,4,6-Tribromophenol	330	332, 141	Acenaphthene-d10	164	162, 160
d-5 Nitrobenzene	82	128, 54	Phenanthrene-d10	188	94, 80
2-Fluorobiphenyl	172	171	Chrysene-d12	240	120, 236
Terphenyl	244	122, 212	Perylene-d12	264	260, 265
2-Chlrophenol-d4	132	68, 134			
1,2-Dichlorobenzene-d4	152	115, 150			

Table 7
Semivolatile Internal Standards with Corresponding TCL Analytes Assigned for Quantitation

1,4-Dichlorobenzene-d4	Naphthalene-d8	Acenaphthene-d10
Phenol	Nitrobenzene	Hexachlorocyclopentadiene
bis(2-Chloroethyl)ether	Isophorone	2,4,6-Trichlorophenol
2-Chlorophenol	2-Nitrophneol	2,4,5-Trichlorophneol
1,3-Dichlorobenzene	2,4-Dimethylphenol	2-Chloronaphthalene
1,4-Dichlorobenzene	Benzoic acid	2-Nitroaniline
Benzyl Alcohol	Bis(2-Chloroethoxy)methane	Dimethyl Phthalate
1,2-Dichlorobenzene	2,4-Dichlorophenol	Acenaphthylene
2-Methylphenol	1,2,4-Trichlorobenzene	2,4-Dinitrophenol
bis(2-Chloroisopropyl)ether	Naphthalene	4-Nitrophenol
4-Methylphenol	4-Chloroaniline	Dibenzofuran
N-nitroso-Di-n-propylamine	Hexachlorobutadiene	2,4-Dinitrotoluene
Hexachloroethane	4-Chloro-3-Methylphenol	2,6-Dinitrotoluene
2-Fluorophenol (surr)	2-Methylnaphthalene	Diethyl Phthalate
Phenol-d6 (surr)	Nitrobenzene-d5 (surr)	4-Chlorophenyl phenyl ether
2-Chlorophenol-d4 (surr)	2,6-Dichlorophenol	Fluorene
1,2-Dichlorophenol-d4 (surr)	α,α-Dimethyl-phenethylamine	4-Nitroaniline
Aniline	o,o,o-Triethyl phosphorothiomine	2-Fluorobiphenyl (surr)
Ethyl methanesulfonate	2,5-Dichlorophenol	2,4,6-Tribromo Phenol (surr)
Methyl methanesulfonate	Hexachloropropene	1-Chloronaphthalene
2-Picoline	p-phenylenediamine	1-Naphthylamine
N-nitrosomethylethylamine	Safrole	2-Naphthylamine
N-Nitrosodiethylamine	1,2,4,5-Tetrachlorobenzene	Pentachlorobenzene
Acetophenone	1-Methylnaphthalene	2,3,4,6-Tetra-chlorophenol
3-Methylphenol	1,2,3,5-Tetrachlorobenzene	2,3,4-Trichlorophenol
N-nitrosomorpholine		Isosafrole
N-nitrosopyrrolidine		1,4-Naphthoquinone
O-toluidine		1,3-Dinitrobenzene
Indene		3,4-Dichlorophenol
2-Chlorophenol-d4 (surr)		2-Methyl-5-nitroaniline
1,2-Dichlorophenol-d4 (surr)		2,3,4,5-Tetrachlorobenzene
		4-Nitroquinoline-1-oxide
Phenanthrene-d10	**Chrysene-d12**	**Perylene-d12**
4,6-Dinitro-2-methylphenol	Pyrene	Di-n-octyl Phthalate
N-nitrosodiphenylamine	Butylbenzyl	Benzo(b) fluoranthene
1,2-Diphenylhydrazine	3,3'-Dichlorobenzidine	Benzo(k) fluoranthene
4-Bromophenyl Phenyl Ether	Benzo(a) anthracene	Benzo(a) pyrene
Hexachlorobenzene	Bis(2-ethylhexyl) Phthalate	Indeno(1,2,3-cd) pyrene
Pentachlorophenol	Chrysene	Dibenz(a,h)anthracene
Anthracene	Terphenyl-d14(surr)	Benzo (g,h,i) pyrene
Di-n-butyl Phthalate	Benzidine	Dibenz(a,j)acridine
Fluoranthene	p-Dimethylaminoazobenzene	Quinoline
4-Aminobiphenyl	3-Methylcholanthrene	7,12-Dimethylbenz(a)anthracene
Diphenylamine	Aramite	Hexachlorophene
Pentachloronitrobenzene	Chlorobenzilate	
Phenacetin	Kepone	
Pronamide	Famphur	
Sulfotep	2-Acetylaminofluorene	
Phorate	3,3'-Dimethylbenzidine	
1,3,5-Trinitrobenzene	6-methyl chrysene	
Diallate		
Dimethoate		
Disulfoton		
2-sec-butyl-4,6-Dinitrophenol		
Carbazole		
Methyl Parathion		
Parathion		
Methapyrilene		
Isodrin		
Benzidine		
Thiophenol		

Typist: LLR

Table 8
Semivolatile Calibration Check Compounds (CCC)

Acenaphthene	Fluoranthene	2-Nitrophenol
1,4-Dichlorobenzene	Benzo(a)Pyrene	Phenol
Hexachlorobutadiene	4-Chloro-3-Methylphenol	Pentachlorophenol
N-Nitroso-Di-n-Phevlamine	2,4-Dichlorophenol	2,4,6-Trichlorophenol
Di-n-Octylphthalate		

Initial calibration 20, 50, 80, 120, and 160ng.

The maximum % RSD for Initial Calibration is 30% for CCC compounds.

Maximum percent difference (or drift) in the continuing calibration is 20% for CCC compounds.

Table 9
Semivolatile System Performance Check Compounds (SPCC)

N-Nitroso-Di-n-Propylamine
Hexachlorocyclopentadiene
2,4-Dinitrophenol
4-Nitrophenol

The Minimum Relative Response Factor/Initial Calibration Average: 0.05, Continuous Calibration: 0.05
All other compounds must meet a minimum response factor of 0.01.

Table 10
Target Compound List (TCL) and Contract Required Quantitation Limits (CRQL) and Practical Quantitation Limits (PQL)

Semivolatiles	CAS Number	Quantitation Limits	
		Water Low ug/L	* Soil/Sediment ug/Kg
1 Phenol	108-95-2	10	330
2 bis(2-Chloroethyl)ether	111-44-4	10	330
3 2-Chlorophenol	95-57-8	10	330
4 1-3-Dichlorobenzene	541-73-1	10	330
5 1,4-Dichlorobenzene	106-46-7	10	330
6 Benzyl alcohol	100-51-6	10	330
7 1,2-dichlorobenzene	95-50-1	10	330
8 2-Methylphenol	95-48-7	10	330
9 bis(2-Chloroisopropyl)ether	108-60-1	10	330
10 4-Methylphenol	106-44-5	10	330
11 N-Nitroso-di-n-dipropylamine	621-64-67	10	330
12 Hexachloroethane	67-72-1	10	330
13 Nitrobenzene	98-95-3	10	330
14 Isophorone	78-59-1	10	330
15 2-Nitrophenol	88-75-5	10	330
16 2,4-Dimethylphenol	105-67-9	10	330
17 Benzoic acid	65-85-0	25	830
18 bis(2-Chloroethoxy)methane	111-91-1	10	330
19 2,4-Dichlorophenol	120-83-2	10	330
20 1,2,4-Trichlorobenzene	120-82-1	10	330
21 Naphthalene	91-20-3	10	330
22 4-Chloroaniline	106-47-8	10	330
23 Hexachlorobutadiene	77-47-4	10	330
24 4-Chloro-3-methylphenol (para-chloro-meta-cresol)	59-50-7	10	330
25 2-Methylnaphthalene	91-57-6	10	330
26 Hexachlorocyclopentadiene	77-47-4	10	330
27 2,4,6-Trichlorophenol	88-06-2	10	330
28 2,4,5-Trichlorophenol	95-95-4	25	830
29 2-Chloronaphthalene	91-58-7	10	330
30 2-Nitroaniline	88-74-4	25	830
31 Dimethylphthalate	131-11-3	10	330
32 Acenaphthylene	208-96-8	10	330
33 2,6-Dinitrotoluene	606-20-2	10	330
34 3-Nitroaniline	99-09-2	25	830
35 Acenaphthene	83-32-9	10	330
36 2,4-Dinitrophenol	51-28-5	25	830
37 4-Nitrophenol	100-02-7	25	830
38 Dibenzofuran	132-64-9	10	330
39 2,4-dinitrotoluene	121-14-2	10	330
40 1,2-Diethylphthalate	84-66-2	10	330
41 4-Chlorophenyl-phenyl ether	7005-72-3	10	330
42 Fluorene	86-73-7	10	330
43 4-Nitroaniline	100-01-6	25	830
44 4,6-Dinitro-2-methylphenol	534-52-1	25	830
45 N-nitrosodiphenylamine	86-30-6	10	330
46 4-Bromophenyl-phenyl ether	101-55-3	10	330
47 Hexachlorobenzene	118-74-1	10	330
48 Pentachlorophenol	87-86-5	25	830
49 Phenanthrene	85-01-8	10	330
50 Anthracene	120-12-7	10	330
51 Di-n-butylphthalate	84-74-2	10	330
52 Fluoranthene	206-44-0	10	330
53 Pyrene	129-00-0	10	330
54 Butylbenzylphthalate	85-68-7	10	330
55 3,3'-Dichlorobenzidine	91-94-1	10	330
56 Benzo(a)anthracene	56-55-3	10	330
57 Chrysene	218-01-9	10	330
58 bis(2-Ethylhexyl)phthalate	117-81-7	10	330
59 Di-n-octylphthalate	117-84-0	10	330
60 Benzo(b)fluoranthene	205-99-2	10	330
61 Benzo(k)fluoranthene	207-08-9	10	330
62 Benzo(a) pyrene	50-32-8	10	330
63 Indeno(1,2,3-cd)pyrene	193-39-5	10	330
64 Dibenz(a,h)anthracene	53-70-3	10	330
65 Benzo(g,h,i)perylene	191-24-2	10	330

NOTE:
Medium Soil/Sediment Contract Required Quantitation Limits (CRQL) for semivolatile TCL Compounds are 60 times the individual Low/Soil/Sediment CRQL. Reporting Limits are from Method.

Specific quantitation limits are highly matrix dependent. The quantitation limits listed herein re provided for guidance and may not always be achievable. Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment, calculated on dry weight basis as required by the contract, will be higher.

Typist: LLR

Table 11
Standards Preparation

Compound		Conc. (µg/mL)	Aliquot (mL)	Total Vol. (mL)	Conc. (µg/mL)	Volume used/µL
Absolute Internal Std. Mix		4000	10	40	1000	—
STOCK STANDARD (Restek)				12		
Methylene Chloride			880			
SV Calibration Mix #1		2000	1200		200	—
SV Calibration Mix #2		2000	1200		200	—
SV Calibration Mix #3		2000	1200		200	—
SV Calibration Mix #4		2000	1200		200	—
SV Calibration Mix #5		2000	1200		200	—
SV Calibration Mix #7		2000	1200		200	—
Benzidines Mn (nsi)		2000	1200		200	—
Benzoic Acid (nsi)		5000	480		200	—
Acid Surr. Mix		7500	320		200	—
B-N Surr. Mix		5000	480		200	—
Pyridine		10000	240		200	—
Aniline		10000	240		200	—
1,2,4,5-Tetrachlorobenzene		10000	240		200	—
2,3,4,6-Tetrachlorophenol		10000	240		200	—
1-methylnaphthalene		10000	240		200	—
Azo Benzene		10000	240		200	—
Stock Std.		200	150		10	—
IS mix	(SSTD020)	1000	60	3	20	—
Stock Std.		200	750		25	—
IS mix	(SSTD050)	1000	120	6	20	—
Stock Std.		200	1200		40	—
IS mix	(SSTD080)	1000	120	6	20	—
Stock Std.		200	900		60	—
IS mix	(SSTD120)	1000	60	3	20	—
Stock Std.		200	1200		80	—
IS mix	(SSTD160)	1000	60	3	20	—

These standards are verified with EPA standards regularly. Every time a standard is made, it is recorded in the current standards logbook, and follows protocol for expirations as provided in SOP SWL-OS-309, "Guide To Semivolatile GC/MS Standard Log".

Table 12
Individual Compound Stock Standards Prep

Compound	Concentration	Aliquot	Total Volume	Final Concentration
Pyridine	NEAT	0.1g	10mL	10,000ug/mL
Aniline	NEAT	0.1g	10mL	10,000ug/mL
1,2,4,5-Tetrachlorobenzene	NEAT	0.1g	10mL	10,000ug/mL
2,3,4,6-Tetrachlorophenol	NEAT	0.1g	10mL	10,000ug/mL
1-Methylnaphthalene	NEAT	0.1g	10mL	10,000ug/mL
Azobenzene	NEAT	0.1g	10mL	10,000ug/mL

Table 13
DFTPP Solution Prep

Compound	Concentration ug/mL	Aliquot mL	Total Volume mL	Concentration ug/mL
DFTPP Solution (Protocol)	2500	0.10	10	25
Phenanthrene-d10	2000	0.125		25
Methylene Chloride		9.775		

* DFTPP Solution from Protocol contains 4,4-DDT, Benzidine and Pentachlorophenol along with DFTPP, each at 2500ug/mL.

Table 14
Calculations

Response Factor and Concentration Calculation

$$RF = \frac{(A_x)\ (C_{is})}{(A_{is})\ (C_x)} \quad \text{and} \quad conc_{RF} = \frac{(A_x)\ (C_{is})}{(A_{is})\ (\overline{RF})} \quad \text{or} \quad conc_{LR} = \frac{(A_x) - b}{M}$$

Where:
- A_x = Area of characteristic ion for the measured compound
- A_{is} = Area of the characteristic ion for the specified internal standard
- C_{is} = Concentration of the internal standard (ng/uL)
- C_{RF} = Concentration of the measured compound (ng/uL) using the average response factor
- $\overline{RF}$ = Average of Five Initial RF's for a compound
- C_{LR} = Concentration of the measured compound (ng/uL) using Linear Regression

Surrogate Recovery:

$$\%\text{Surrogate Recovery} = \frac{Qd}{Qa} \times 100\%$$

Where:
- Qd = Quantity determined by analysis
- Qa = Quantity added to sample

Matrix Spike recovery and RPD:

$$\text{Matrix Spike Recovery} = \frac{(SSR - SR)}{SA} \times 100 \qquad \text{Matrix Spike RPD} = \frac{(MSR - MSDR)}{(1/2)\ (MSR + MSDR)} \times 100$$

Where:
- SSR = Spike Sample Result
- SR = Sample Result
- SA = Spike Added

Laboratory Control Spike (LCS) Recovery:

$$\text{LCS Spike Recovery} = \frac{(SSR - SR)}{SA} \times 100$$

Where:
- SSR = Spiked Sample Result
- SR = Sample Result
- SA = Spike Added

Calibration Calculations:

$$\%RSD = \frac{SD}{\overline{RF}} \times 100\% \qquad \%Drift = \frac{(C_1 - C_C)}{C_1} \times 100$$

Where:
- SD = Standard Deviation
- $\overline{RF}$ = Average of Five Initial RF's for compound
- RSD = Relative Standard Deviation

Where:
- C_1 = Calibration Check Compound Standard Concentration
- C_C = Measured concentration using selected quantitation method

Form I

RE-EXTRACT TRACKING SHEET

	CLIENT	EPISODE/ LAB ID #	H20	SOIL	INST.	MS CODE	DATE RCVD	INITIAL EXT. DATE	RE-EXT. SUB. DATE	REASON FOR RE-EXTRACT			RE-EXT. DATE	RE-EXTRACT RESULTS			W/HOLD TIME Y/N
										ACID	B/N	QC		ACID	B/N	QC	
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Attachment 1
Re-extract Tracking Sheet

DATA REVIEW SHEET
SWOK/AATS
ref. SWL-OS-303

ANALYST REVIEW

EPISODE _____ INSTRUMENT _____
MS CODE _____ ANALYST _____

_____ Continuing Calibration updated with correct Method and Initial Calibration
_____ Correct Sublist used for requested compounds
_____ Sample Line correct (sample ID *swlo #) (SBLK *BL0803WA) (DFTPP*DFTPP*1-018-18)
_____ Misc. Line correct (lcip blk*Client Name*Dilution run string) add * %moist. To end if needed
_____ SBLK passed QC criteria (no contamination greater than CRQL, all surrogates and Int. Stds. okay)
_____ Internal Standard areas and retention times checked
_____ Surrogate recoveries checked
_____ Compound linearity checked (note: If linearity is above 1000ng on column, was following sample reviewed for carryover and re-analyzed if necessary?)
_____ Target compounds checked for proper integration
_____ Library Search Compounds (LSC) included for applicable samples
_____ All applicable reports printed and included
_____ % RPD's listed for LCS/LCSD and MS/MSD for non-CLP data
_____ If any re-extracts were performed, are remedial action forms attached? Yes_____ No_____

Analyst comments _____

DATA REVIEW

_____ False Positives deleted form system
_____ LSC Edited
_____ Holding Times met for extraction_____ analysis_____
_____ Any Dilutions or Re-analysis checked against original sample
_____ LCS / LCSD pass
_____ MS / MSD pass and matches original non-spiked sample

Initial Data Review by _____ Date ____/____/____
Final Data Verification by_____ Date ____/____/____
Data Uploaded to LIMS_____ Date ____/____/____
Reviewer Comments_____

Attachment 2
Data Review Sheet

NON-CONFORMANCE REPORT QA DEPT.

SWL-GA-105 GA-105-NCR-F SWL Rev. No.:_____

Department:

8.2.1.2 DESCRIPTION OF NON-CONFORMANCE

Initiated By: Date:

Section Supervisor: Date:

Date of Occurrence:

Description of Event:

System and/or Episode of Samples Affected (Client/Test code/Matrix if applicable):

8.2.1.3 CAUSE

Description:

SIGNATURE:

Section Supervisor: Date:

8.2.1.3 ACTION TO BE TAKEN

Re-preparation/ Re-extraction Required (Yes/ No)? Date Needed:

Within hold time: (Yes/No)? Sample Receipt Date (if applicable):

Description (with dates):

SIGNATURE:

Completion Date: Section Supervisor:

SOUTHWEST LABORATORY OF OKLAHOMA, INC.
1700 W. ALBANY• BROKEN ARROW, OK 74012 •OFFICE (918) 251-2858 •FAX (918) 251-2599

Attachment 3
Non-Conformance Report

SAMPLE EXTRACTION LOG

SWOK • AATS

Southwest Laboratory of Oklahoma, Inc.
American Analytical & Technical Services, Inc.

☐ RUSH ☐ REEXTRACT ☐ TCLP ☐ DIOXIN

CLIENT: _____ DATE RECEIVED: _____

CASE: _____ EXTRACT DATE: _____

METHOD	SV	PEST • PCB	OTHER
CONT. EX.			
SEP-F			
SONC.			

SAMPLE ID (EPISODE #)	CLIENT ID	MATRIX - LEVEL	SAMPLE pH	% MOISTURE	DRY WEIGHT	AMOUNT EXTRACTED	EXTRACTED VOLUME	GPC VOLUME	GPC ELUTE VOL	FLORISIL VOL CONC	FLORISIL FM VOL CONC	VOL SV - P	FM PROCESS VOLUME	SCREEN T/N	MS SV - P	SURR SV - P	BASE pH CONFIRMATION	ACID pH CONFIRMATION	COMMENTS
				%	g	g • mL	mL	mL	mL	mL	mL	mL	mL		µL	µL			

INITIALS	
WEIGHING:	EXTRACTION:
CLEANUP:	KD:
BLOWDOWN:	
SURROG. SPIKER:	VERIFIER:
MATRIX SPIKER:	VERIFIER:
SUBMITTED BY	REVIEWED BY:

ID NUMBERS
FLORISIL CARTRIDGE #:
GPC COLUMN #:
SURROGATES ID CONC #:
MATRIX SPIKE SOL ID/CONC #(S):
MISC:

COMMENTS

[DX013-0393-01]

54201

Attachment 4
Sample Analysis/Extraction Log

EXTRACT RECEIPT LOG

SEMIVOLATILE GC/MS LABORATORY

DATE	TIME	SWOK / AATS SAMPLE ID	CLIENT ID	EXTRACTION CHEMIST INITIALS	GC/MS SAMPLE CUST. INITIALS	COMMENTS	NO. OF SAMPLES

SOUTHWEST LABORATORY OF OKLAHOMA, INC. • AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.
1700 W. ALBANY • BROKEN ARROW, OKLAHOMA 74012 • OFFICE (918) 251-2858 • FAX (918) 251-2599

[SGM005-0193-02]

Attachment 5
Extract Receipt Log

GC/MS SEMIVOLATILE RUN LOG SWOK/AATS SOP: SWLO-OS-302 Rev. 2.0 START DATE: 20 Jan 2000

DATAFILE PATH: G:\HPCHEM\F\DATA\F_0120\ INT. STD #: 1-043-19 INST: F EMV: 1400

ANALYSIS METHODS: DFTPPF2 ,8270C2 PROCESS METHODS: temp.m,F8460103.M

EPISODE: 41748 MSCODE : MS599 TUNE FILE ID : TUNEF

TIME	INIT	CLIENT SAMPLE ID	LAB ID	FILE ID	SAMPLE AMOUNT WT/V	EXT	INJ	DIL FACT	IS AREA	SUR REC	LIN CHK	COMMENT
10:42	AEW	SOLVENT BLANK	DFTPP	F0012001			2UL	1	N/A	N/A	N/A	
11:04	AEW	SOLVENT BLANK		F0012003			2UL	1				
11:15	AEW	DFTPP	DFTPP	F0012004	N/A	N/A	2UL	1	N/A	N/A	N/A	SW-OK
11:39	AEW	SSTD080	SSTD080	F0012005	N/A	N/A	2UL	1	N/A	N/A	N/A	✓
12:12	AEW	SSTD080	SSTD080	F0012006	N/A	N/A	2UL	1	N/A	N/A	N/A	No Use
12:54	AEW	SSTD160	SSTD160	F0012007	N/A	N/A	2UL	1	N/A	N/A	N/A	✓
13:27	AEW	SSTD020	SSTD020	F0012008	N/A	N/A	2UL	1	N/A	N/A	N/A	✓
14:00	AEW	SSTD120	SSTD120	F0012009	N/A	N/A	2UL	1	N/A	N/A	N/A	✓
14:33	AEW	SSTD050	SSTD050	F0012010	N/A	N/A	2UL	1	N/A	N/A	N/A	✓
15:06	AEW	SSTD080 2nd source	SSTD080	F0012011	N/A	N/A	2UL	1	N/A	N/A	N/A	ok
15:39	AEW	SBLK	BL0118WD	F0012012	1000	1ML	2UL	1	✓	✓	✓	✓
16:12	AEW	LCS	LC0118WD	F0012013	1000	1ML	2UL	1	✓	✓	✓	✓
16:44	AEW	LCSD	LD0118WD	F0012014	1000	1ML	2UL	1	✓	✓	✓	✓

NA=Not Available N/A=Not Applicable F=Failed

Analyst Signature: Ann E. Wade Reviewed by: Diana K. Hoh

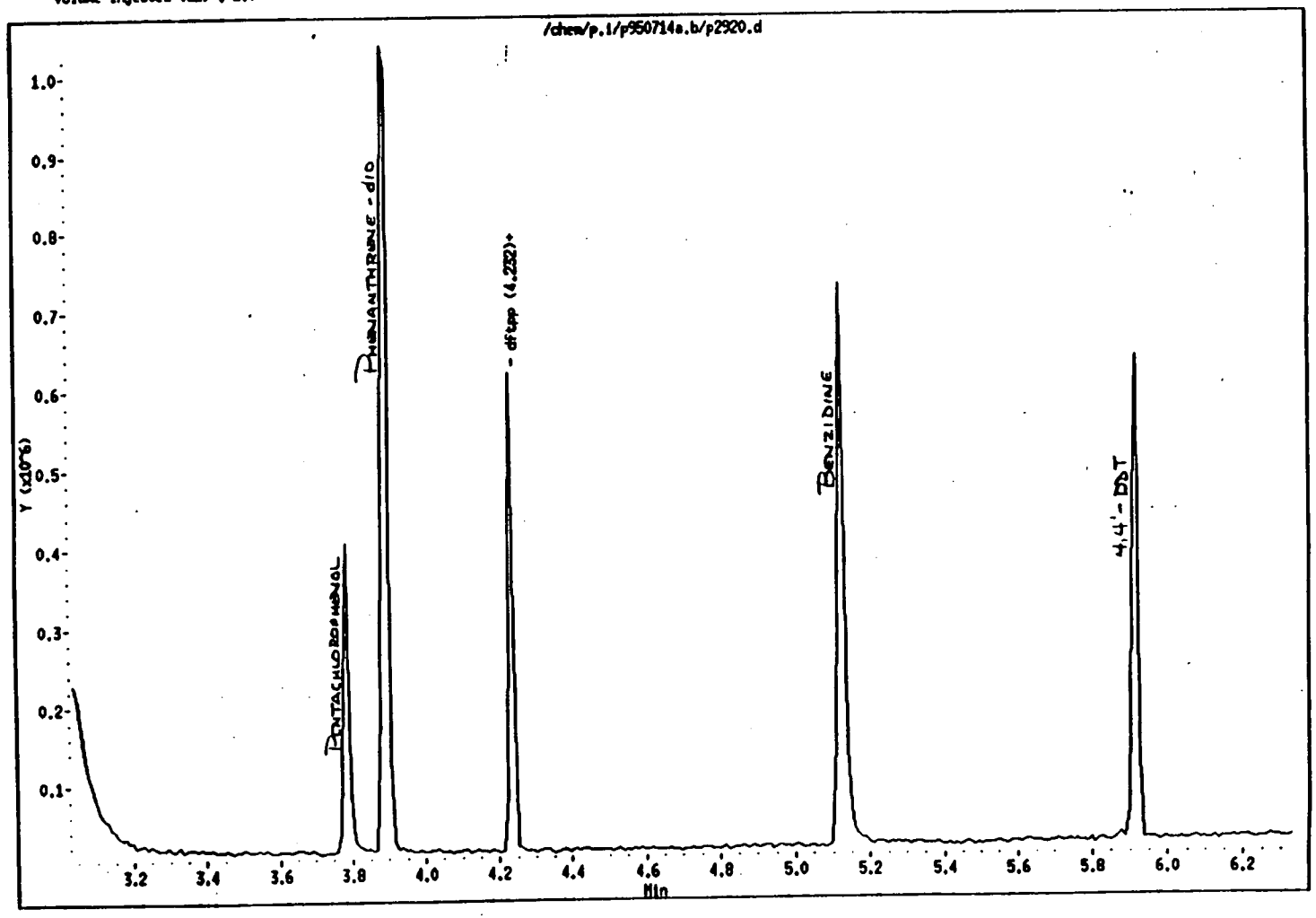
Book : OS-302-RUN-F9 Page : 2

Attachment 6
Semivolatiles GC/MS Run Log

Data File: /chem/p.1/p950714a.b/p2920.d
Date : 14-JUL-95 14:25
Instrument : p.1
Sample ID : DFTPP
Column phase : XTI-5
Volume Injected (uL) : 2.0

Column diameter : 0.25

Page 1

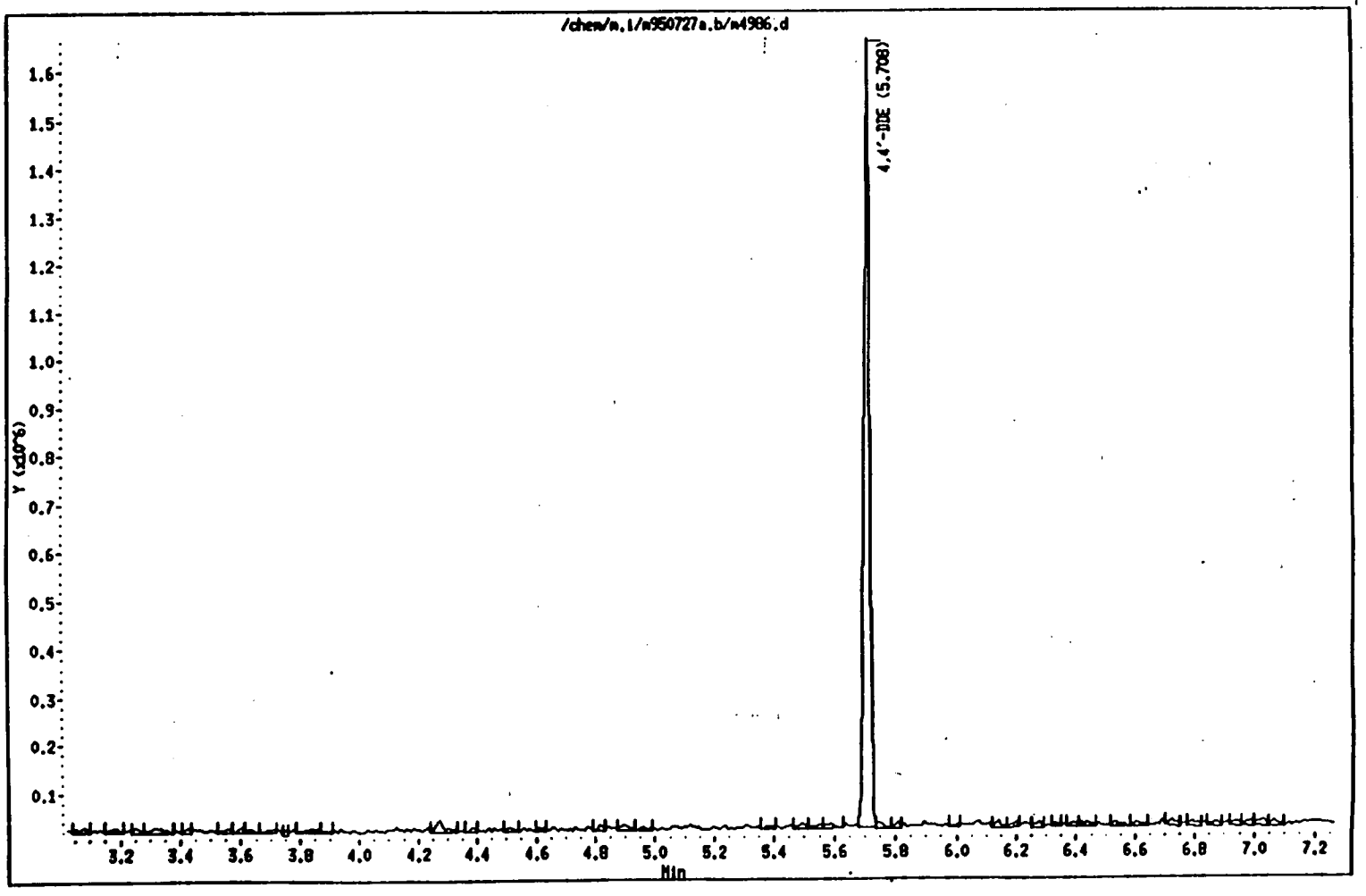


Attachment 7
DFTPP (Page 1 of 3)

Data File: /chem/m.i/m950727a.b/m4986.d
Date : 27-JUL-1995 16:37
Instrument : m.i
Sample ID : 4,4'-DDE
Column phase : XTI-5
Volume Injected (uL) : 2.0

Page 1

Column diameter : 0.25

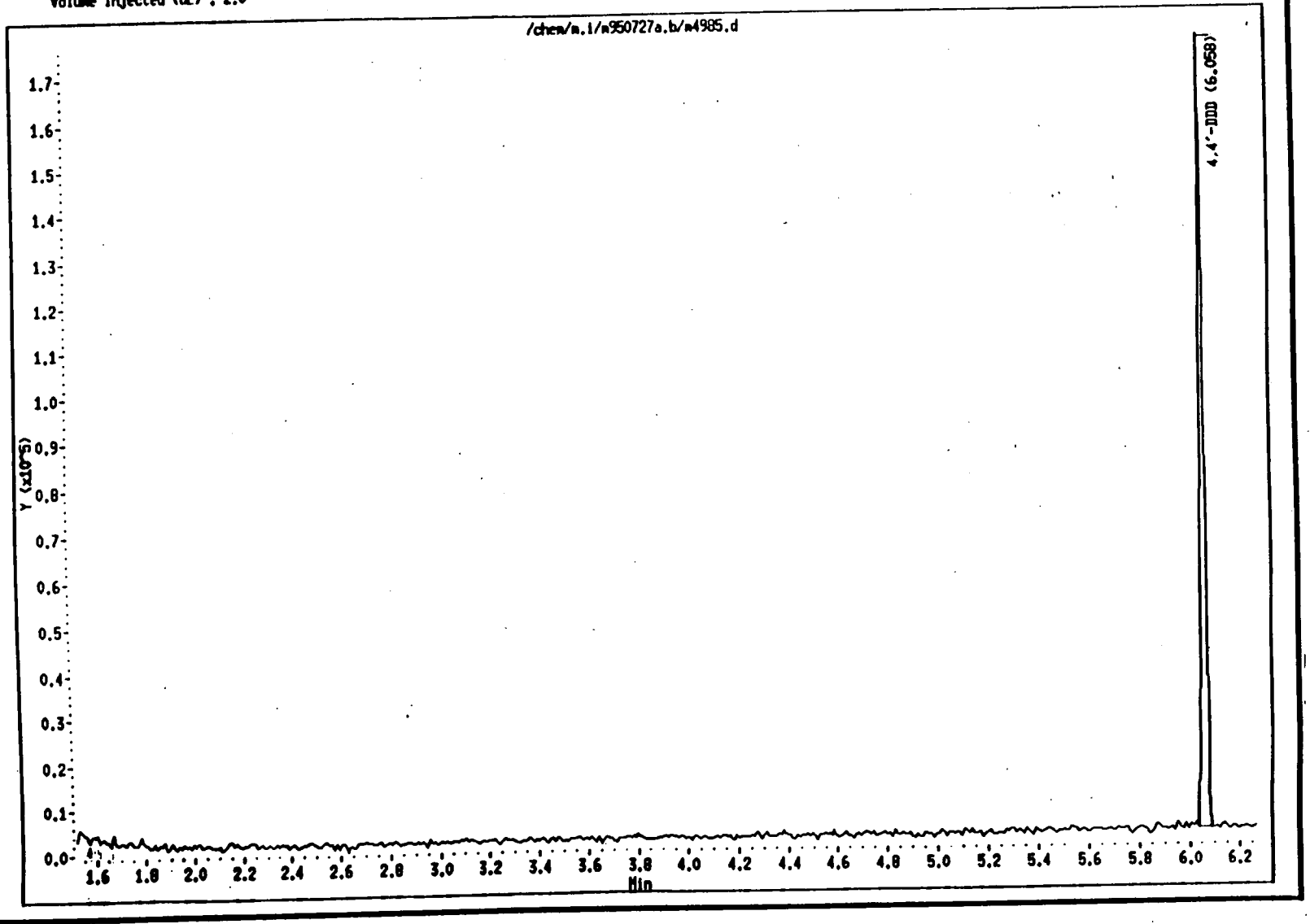


Attachment 8
DFTPP (Page 2 of 3)

Page 1

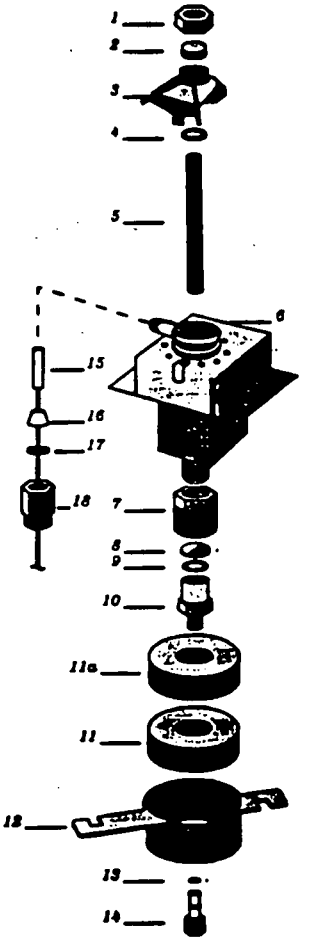
Data File: /chem/m.i/m950727a.b/m4985.d
Date : 27-JUL-1995 16:27
Instrument : m.i
Sample ID : 4,4'-DDD
Column phase : XTI-5
Volume Injected (uL) : 2.0

Column diameter : 0.25



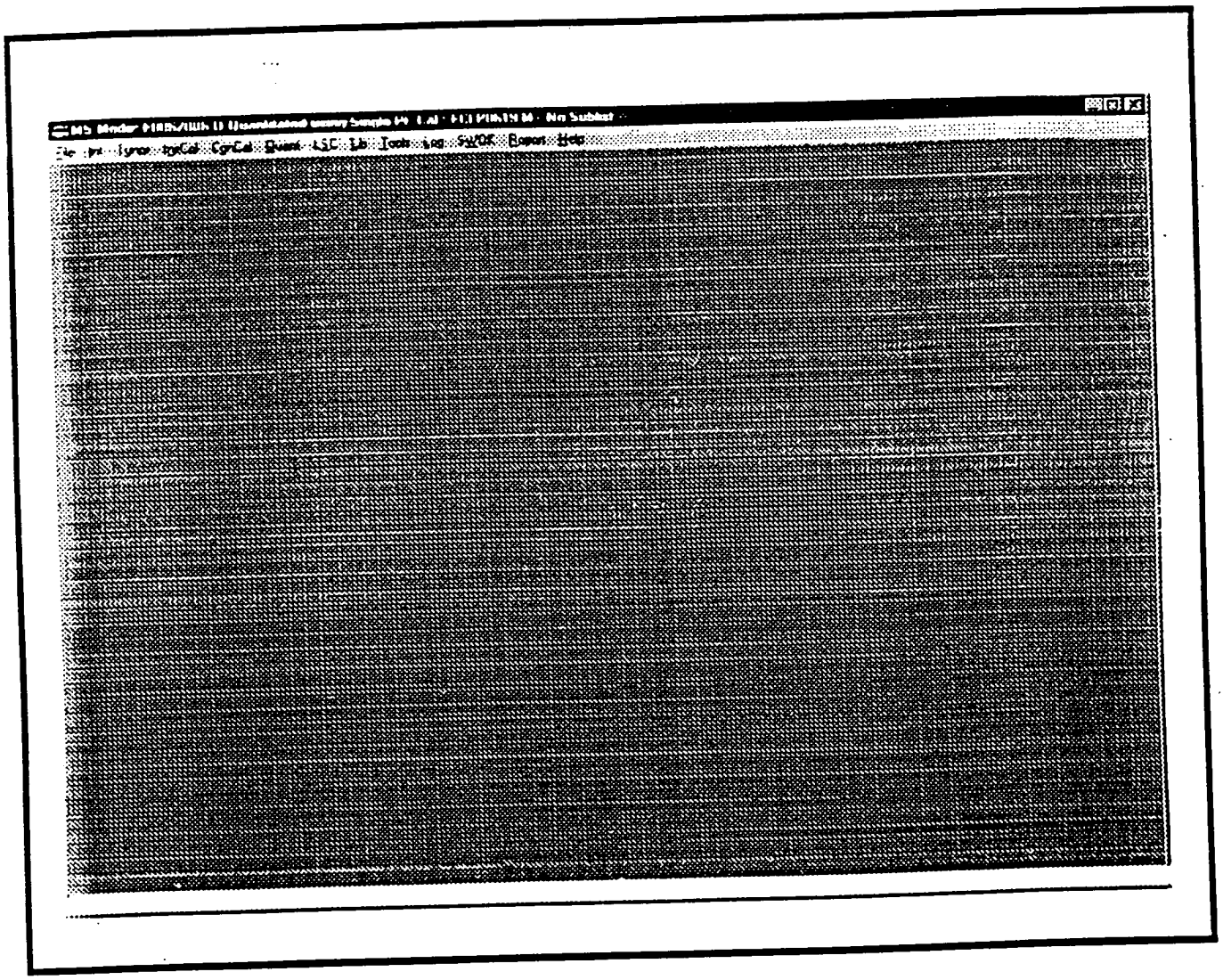
Attachment 9
DFTPP (Page 3 of 3)

1	[illegible]	[illegible]
	[illegible] retainer nut for Headspace [illegible]	[illegible]
	[illegible] for Merlin Microseal [illegible] (see page [illegible])	
2	Septa (see page 50)	[illegible]
3	Split/splitless insert Weldmark with [illegible]	[illegible]
4	Liner seals (also refer to liner table on page [illegible])	
	a. Viton O-ring for high temperature (max 350 °C) (12/pk)	5180-1182
	b. Graphite O-rings (temperature above 350 °C)	
	For split liner (12/pk)	5180-1184
	For splitless liner (12/pk)	5180-4173
5	See liner table on page 42	
6	Shell weldment (refer to instrument manuals)	
7	Retaining nut	19251-20620
8	Gold-plated seal	19740-20885
	or stainless steel seal	19740-20880
9	Washer (12/pk)	5061-5889
10	Reducing nut (1 ea)	19740-20800
11	Lower insulation	19243-00060
11a	Insulation	19243-00065
12	Lower insulation cover	19243-00070
13	Column seals/ferrules—page 44	[illegible]
14	Column nut (2/pk)	5181-8830
15	Splitter tube	19251-60525
16	Front ferrule, X-in. brass (10/pk)	5180-4109
17	Back ferrule, X-in. brass (10/pk)	5180-4115
18	Tubing nut, X-in. brass (10/pk)	5180-4103



Split/Splitless (Multimode) Inlet

Attachment 10
Split/Splitless (Multimode) Inlet



Attachment 11
Enviroquant Window

Typist: LLR

FORM 1

BACK-UP BLANK USAGE FORM

When a back-up blank is used this form must be completed. The signed original must be retained with the original data and a copy given to the department supervisor.

Primary Blank ID: BL_____ Date Analyzed:_____

Primary Blank Data file ID:_____ Analyst:_____

Supervisor:_____

Back-up Blank ID: BB_____ Date Analyzed:_____

Back-up Blank Data file ID:_____ Pass or Fail (circle one)

Matrix: Water Soil Other_____ Analyses Code: MS_____

GC_____

Reasons for Back-up Blank Usage: (Check all that apply)

____Surrogates Failed: ____ Acid Surrogates ____ Base/Neutral Surrogates

____Internal Standards Failed: ____Out High ____Out Low

____Contamination (Target Analyte):____Above reporting limit ____Trace Amounts

____Extract Lost: ____Vial Broken ____Extract Spilled ____Other (explain)

_____

_____

Samples Affected:_____

_____

_____

If both blanks fail, note Remedial or Corrective (circle one) Action Taken:_____

_____

Attachment 12
Back-up Blank Usage Form

SOUTHWEST LABORATORY OF OKLAHOMA, INC. *and*
AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.

Standard Operating Procedure

Volatile Data Review

Document No.: SWL-OV-103

Rev No. / Date: Rev. No.: 5.0 — 10/06/00

OFFICIAL COPY RED STAMP INDICATES ORIGINAL

APPROVALS	DATES
James Whittaker Procedure Prepared By	10/11/00 Date
Hilary In Bre Program Manager or Section Supervisor	10-24-00 Date
Bryan Hach Laboratory Safety Officer	10/24/2000 Date
Chuck Hoover Laboratory QA/QC Officer	10/24/00 Date

(Effective Date is 5 calendar days after the last signature above - QA/QC Officer)

Document Status

[ ] Controlled DC No. _____ Issued to: _____ Date: _____ QA: _____

[✓] Official Copy OC No.: 2431 Issued to: Linda Karasonovich Date: 11/14/00 QA: [illegible]

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VOLATILE DATA REVIEW

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1.0 SCOPE AND APPLICATION

1.1 The purpose of this standard operating procedure is to provide guidelines for volatile data review. It will provide specific instructions to ensure the review process is performed with consistency.

1.2 Data review may include, but is not limited to, reviewing instrument calibrations and raw data, reviewing sample report forms, downloading data into the Volatile Departmental Database, and verifying files within this database.

1.3 The Volatile Section Supervisor or Volatile Data Reviewer will be responsible for the final data review within the Volatile Department. All Volatile GC/MS Analysts are responsible for initial review of the calibrations and raw data in which they generate. Volatile Data Clerks are responsible for initial review of all forms and diskettes generated for client reports within the Volatile Department (i.e.: CLP forms, Data Package forms, and EPA diskettes).

2.0 METHOD SUMMARY

Not applicable.

3.0 HEALTH AND SAFETY

Not applicable.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

Not applicable.

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

Not applicable.

6.0 EQUIPMENT/APPARATUS

6.1 Volatile Departmental Database (DDB)

6.2 Enviroquant Chemstation, G1701AA Version A.03.00.

7.0 REAGENTS

Not applicable.

8.0 PROCEDURE

8.1 Calibration Review

8.1.1- Each analyst performs a daily instrument calibration before any samples are analyzed. These calibrations are immediately turned into the Volatile Section Supervisor or Data Reviewer for verification the calibration is acceptable. Both the analyst (during an initial review) and the Supervisor/Data Reviewer (during a final review) perform the following steps:

8.1.1.1 BFB-Bromofluorobenzene

- Initial and date the top right-hand corner of the BFB chromatogram.
- Review the tune ratios to verify all meet criteria. Any ratios outside limits will be flagged.

8.1.1.2 Standard Calibration

- Initial and date the top right-hand corner of the Standard(s) chromatogram.
- Review the Initial Calibration Report, Continuing Calibration Report, or Calibration Verification Report to verify all response factors meet specifications (ie: RRF, %RSD, % Difference, % Drift, Linear Regression Coefficients.)
- Check the Initial Calibration Date and Method ID to verify calibration information was updated properly.
- Check the chromatogram(s) to ensure no carryover exists that could affect the response of one or more compounds.
- Check the retention times to verify no compounds are coeluting. Below are some common checkpoints and the order they elute from left to right:

  1. Vinyl Acetate and 2-Butanone
  2. Tetrachloroethene and Dibromochloromethane
  3. Ethylbenzene, m,p-Xylene, and o-Xylene
  4. 1,3-Dichlorobenzene, 1,4-Dichlorobenzene, and 1,2-Dichlorobenzene
  5. tert-Butylbenzene, sec-Butylbenzene, and N-Butylbenzene
  6. 1,2,4-Trichlorobenzene and 1,2,3-Trichlorobenzene
  7. 1,3,5-Trimethylbenzene and 1,2,4-Trimethylbenzene
  8. 2-Chlorotoluene and 4-Chlorotoluene

- Verify all manual integrations have been submitted, initialed, and dated.

8.1.1.3 Method Blank

- Initial and date the top right hand corner of the chromatogram of the Method Blank.
- Review the chromatogram to make sure it is free of carryover/contamination.
- Check the quant report and delete any "False Positives" by drawing a line through the compound and labeling the line with "FP". Stamp the quant report at the bottom with the "False Positive" stamp and initial and date. Also, pull the spectrum out corresponding to the False Positive.
- Verify the method blank used the proper calibration file, which is listed, on the quant report page under "ICAL Path."

8.1.1.4 Laboratory Control Spike/Duplicate (LCS/LCSD)

- Initial and date the top right-hand corner of the chromatogram of both the LCS and/or LCSD.
- Review the quant report and check retention times for coelution of compounds.
- Verify the proper compounds were spiked in the LCS/LCSD analyses.
- Check the Spike Recovery Report noting any compounds outside recovery limits. These recovery limits are advisory, but since they are internal QC limits set forth by our laboratory, every effort should be made to meet these limits.

8.1.2 If any of the above items do not meet criteria, the analyst must perform corrective action. If any samples were analyzed under a defective calibration, they must be reprocessed, reanalyzed, or the deficiency must be noted by the Supervisor/Data Reviewer in the Case Narrative.

8.1.3 Calibrations and their corresponding samples denote a "batch". They are downloaded to the Volatile Departmental Database following the instructions in Section 8.6 after the complete batch has been processed. An Injection Log (Attachment 1) displaying pertinent information about the batch is printed at this time and reviewed.

8.1.4 This Injection Log is attached to the front of the original calibration, and submitted to the Volatile Data Clerk for copying and filing. The tune, calibration, method blank, LCS, and LCSD are downloaded into the Volatile Departmental Database upon completion of processing.

8.2 Analyst Review

8.2.1 After the instrument calibration is performed and deemed acceptable. sample analysis may begin. Each analyst reviews the following parameters for every analysis performed.

8.2.1.1 *Internal Standard (I.S.) Areas and Retention Times*: Check the I.S. Check Report to make sure Internal Standards are within the method's specified limits. Take appropriate action when necessary. Also, verify the correct areas from the calibration standard were updated into the appropriate method.

8.2.1.2 *Surrogate Recoveries*: Check the Surrogate Recovery Report for flags showing surrogates outside specified QC Limits. Take appropriate action when necessary.

8.2.1.3 *Target Compounds:* Check the quant report for positive target compound hits that may be above the linear range and require a dilution. If a compound is detected above the linear range, determine what level of dilution is required. In addition, monitor the sample analyzed immediately after the contaminated sample for all target compounds that were above the linear range. The sample must not contain a concentration above the Quantitation Limit to be acceptable: otherwise it must be reanalyzed. If the sample immediately following the contaminated sample does contain those target compounds above the Quantitation Limit, the next sample must be monitored for the same criteria, and so on.

8.2.1.4 *Enviroquant Review:* Step through each target compound, checking for proper integration and isomeric compound separation. Delete any obvious false positives. If there is any question or doubt concerning false positives, leave for review.

8.2.1.5 *Sample Information:* While in Qedit or by reviewing the hardcopy of the quant report, review the following parameters:

- *Compound Sublist*: Refer to Episode Sheet for correct sublist
- *Sample:* CLIENTID*SWLOID
- *Misc. Info Line:* *CLIENT*DILUTION STRING

8.2.1.6 When review is complete and sample info has been edited, generate the appropriate reports.

8.2.1.7 Assemble the data in the following order and submit to the Data Clerks:

1. Episode Sheet
2. Sample Report (if Standard Tabular or if preliminary data is required.)

3. Chromatogram
4. Quant Report
5. Manual Integrations (if any), initialed and dated
6. Mass Spectral Data for each positive target compound hit (with reference spectra
7. Tentatively Identified Compound Spectra
8. Internal Standard Area and RT Summary
9. Surrogate Spike Recovery Report
10. Reanalysis (when applicable)
11. Extra Data

8.3 Initial Review of Data

8.3.1 Initial and date the top right-hand corner of the chromatogram of the sample to be reviewed.

8.3.2 Follow step 8.2.1.3 of Analyst Review, checking all positive target hits for false positives and false negatives and need for dilutions.

8.3.3 Follow the steps contained in 8.1.1.3 for labeling False Positives.

8.3.4 Make sure all appropriate actions were taken for samples not meeting QC Requirements. For example:

- Sample reanalyzed if surrogate(s) are outside Surrogate Recovery Limits
- Sample reanalyzed if Internal Standard(s) are outside QC Area or Retention Time Limits
- Sample analyzed at a dilution when target analytes are above the linear range
- Sample reanalyzed if original sample was injected outside calibration clock

8.3.5 Review all TICs and edit their identification under "Edit LSC Results."

8.3.6 Glance through Extra Data and verify any reanalyses correspond to the original, and that everything in Extra Data is actually unreportable data.

8.3.7 If there are any discrepancies or mistakes in the data, return the case to the analyst for correction and explain the problem.

8.3.8 If data is to be reported in a Standard Tabular Report format, proceed with Section 8.4. If data is to be reported as a Data Package, return package to Data Clerks for forms generation and review final data package with forms as per Section 8.5.

8.4 Final Review/Verification of Standard Tabular Report Data

8.4.1 After files are reviewed and edited. make any corrections to the sample. print out a clp.rp report and attach to the front report.

8.4.2 Upload all sample files to the Volatile Departmental Database. following the steps in Section 8.6

8.4.3 Verify the samples uploaded correctly with the proper test code by reviewing their "Sample Status" on the Intranet.

8.4.4 Submit a Case Narrative describing analysis methods. QC, Sample Information. and any problems incurred. Place the narrative on the front of the samples.

8.5 Final Review/Verification of Data Packages and CLP Packages

8.5.1 Pull pH log to front of package to be placed directly behind the Case Narrative (Data Package Format-Attachment 4) or (CLP Format-Attachment 5).

8.5.2 Check Episode Sheet to verify the correct forms were generated.

8.5.3 Verify "Heated Purge" is correct on all forms. May be correct on Form V, but not on Form VIII.

8.5.4 Verify the Sample Form I contains the correct compound list and that is contains the same number of target compounds hits as the quantitation report.

8.5.5 Check the Initial Calibration and Continuing Calibration (or Calibration Verification) to verify the calibration levels are correct on the forms.

8.5.6 Verify the Initial Calibration and Continuing Calibration (or Calibration Verification) Forms contain the correct date and times for the Initial Calibration.

8.5.7 Check to make sure the raw data for all BFB tune analyses are present.

8.5.8 Prepare the Case Narrative including the items listed below:

1. Note Client, SDG, # of samples (excluding MS/MSD) submitted, and the method used during analysis.
2. Note Surrogate Recoveries, specifically listing any samples with recoveries outside QC Limits.
3. Note Matrix Spike/Matrix Spike Duplicate (MS/MSD) and Laboratory Control Spike/Laboratory Control Spike Duplicate (LCS/LCSD), specifically commenting on the number of compounds outside QC Limits.
4. Note Internal Standard Area Recoveries, specifically listing any samples with recoveries outside QC Limits.
5. Note Method Blank, Instrument Blank, Storage Blank, and Holding Blank information, specifically commenting on any contamination.
6. Note any other general information including dilutions, matrix problems, etc.

8.5.9 Verify all samples uploaded correctly to the database during forms generation.

8.6 Uploading Files to Departmental Database for Calibration QC and Standard Tabular Reports.

8.6.1 All standard tabular report samples and calibrations are uploaded through LIMS to the Volatile Departmental Database (DDB). They are uploaded in the following manner:

- In Enviroquant. click on "Report."
- Click on "Upload Files to Database."
- Select the data file(s) to be uploaded.
- Click on "Process."
- A pop-up box will appear requesting LIMS ID and password. A message will then appear in the green line stating whether the upload was complete.

8.7 Uploading Files to Departmental Database for Data Packages

All samples contained in a data package are uploaded to the LIMS during Forms Generation. After the forms are reviewed, they are uploaded in the following manner:

- In the GC/MS Forms Generation Program, click on "Forms."
- Click on "Upload Files to LIMS."
- Enter the sample(s)' test code.
- A pop-up box will appear requesting LIMS ID and Password.
  A message will then appear in the green line indicating whether the upload was complete.

8.8 After files have been uploaded, verify all information is in the database. This information may be checked in the following manner:

- Click on the Intranet
- Click on "LIMS Status"
- Click on "Enter"
- Click on Sample Status
- Click on "By Sample ID"
- Enter the SWLO Episode and click on "Query"
- Click on SWLO Sample ID
- Click on applicable test code

- Click on SWLO Sample ID to check results
- Click on batch ID to verify all applicable QC is uploaded
- Review the Dept. Analyses to verify the samples were uploaded with the proper test code.

9.0 CALCULATIONS

Not applicable.

10.0 QUALITY ASSURANCE/QUALITY CONTROL

Not applicable.

11.0 WASTE DISPOSAL/POLLUTION PREVENTION

Not applicable.

12.0 METHOD PERFORMANCE

Not applicable.

13.0 REFERENCES

Not applicable.

14.0 DEFINITIONS

14.1 Terminology

DDB:.....Volatile Departmental Database: Repository for the storage of volatile data, verification of data, and subsequent sample reporting.

Case Narrative:.....Portion of the data package that includes sample episode or SDG information, and descriptive documentation of any problems encountered in analyzing or processing

SOP:.....Standard Operating Procedure: Written narratives that describe systematic instructions and procedures specific to the laboratory.

Injection Log:.....Summary of instrument sample batches printed from Enviroquant. Summary includes file name, sample name, sample wt/vol., date and time of analysis.

Batch:.....Daily analytical sequence consisting of the daily tune, calibration, method blank, QC spikes, and samples analyzed within a 12-hour time clock.

Extra Data: .....Data classified as non-reportable, but stored by the laboratory with the pertinent episode information for reference purposes.

Episode Sheet:.....Information generated in Sample Receiving as samples are logged into the LIMS. Sheet is specific to each set of samples and contains, at a minimum, the Client, Project, Client ID, SWLO ID, Date Received, Test Required and Number of Containers.

Standard Tabular Report: Type of data package report generated by the Reporting Department from information.

14.2 Acronyms

CLP .....Contract Laboratory Program

LCS/LCSD.....Laboratory Control Sample/Laboratory Control Sample Duplicate

LIMS .....Laboratory Information Management System

RT .....Retention Time

MS/MSD.....Matrix Spike/Matrix Spike Duplicate

TIC.....Tentatively Identified Compound

5.0 ATTACHMENTS

15.1 Attachment 1: Injection Log

15.2 Attachment 2: Case Narrative (Data Package Format)

15.3 Attachment 3: Case Narrative (CLP Format)

ATTACHMENT 1
Injection Log

(Example Only)

Injection Log

Directory: v:\hpchem\k\data\k_1004

Line	Vial	FileName	Multiplier	SampleName	Misc Info	Injected
1	99	k33621.d	1.	BFB *BFB *2-116-9-A	**1UL	4 Oct 00 11:47
2	1	k33622.d	1.	VSTD010 *VSTD010 *2-110-9-A	**25ML	4 Oct 00 11:54
3	2	k33623.d	1.	VBLK *K001004A	**25ML	4 Oct 00 12:44
4	3	k33624.d	1.	LCS *LCS *2-132-9-A	**25ML	4 Oct 00 13:11
5	4	k33625.d	1.	LCSD *LCSD *2-132-9-A	**25ML	4 Oct 00 13:38
6	5	k33626.d	1.	ETI-R1E-S-01-092700MS *44515.13MS	*TETRACO *25ML	4 Oct 00 14:06
7	6	k33627.d	1.	ETI-R1E-S-01-092700MSD *44515.14MSD	*TETRACO *25ML	4 Oct 00 14:33
8	7	k33628.d	1.	ETI-R1E-S-01-092700 *44515.10	*TETRACO *25ML	4 Oct 00 15:00
9	11	k33629.d	1.	ETI-R10-S-01-092700 *44515.01	*TETRACO *12.5ML/25ML	4 Oct 00 15:28
10	12	k33630.d	1.	ETI-R10-S-02-092700 *44515.02	*TETRACO *10ML/25ML	4 Oct 00 15:55
11	13	k33631.d	1.	ETI-R10-S-03-092700 *44515.03	*TETRACO *10ML/25ML	4 Oct 00 16:22
12	18	k33632.d	1.	ETI-R11-S-01-092700 *44515.04	*TETRACO *12.5ML/25ML	4 Oct 00 16:50
13	1	k33633.d	1.	LCSD *LCSD *2-132-9-A	**25ML	4 Oct 00 17:17
14	19	k33634.d	1.	ETI-R11-S-02-092700 *44515.05	*TETRACO *12.5ML/25ML	4 Oct 00 17:45
15	20	k33635.d	1.	ETI-R11-S-03-092700 *44515.06	*TETRACO *12.5ML/25ML	4 Oct 00 18:12
16	21	k33636.d	1.	ETI-R12-S-01-092700 *44515.07	*TETRACO *25ML	4 Oct 00 18:39
17	22	k33637.d	1.	ETI-R13-S-01-092700 *44515.08	*TETRACO *25ML	4 Oct 00 19:06
18	11	k33638.d	1.	ETI-R14-S-01-092700 *44515.09	*TETRACO *25ML	4 Oct 00 19:34
19	12	k33639.d	1.	ETI-R10-T-01-092700 *44515.11	*TETRACO *25ML	4 Oct 00 20:01
20	13	k33640.d	1.	ETI-R10-F-01-092700 *44515.12	*TETRACO *25ML	4 Oct 00 20:28
21	11	k33641.d	1.	002 SW *44470.02	*B&MC *25ML	4 Oct 00 20:55
22	12	k33642.d	1.	003 SW *44470.03	*B&MC *25ML	4 Oct 00 21:23
23	13	k33643.d	1.	004 SW *44470.04	*B&MC *25ML	4 Oct 00 21:50
24	14	k33644.d	1.	TB TB *44470.05	*B&MC *25ML	4 Oct 00 22:17
25	15	k33645.d	1.	ETI-R12-S-01-092700 *44515.07	*TETRACO *25ML	4 Oct 00 22:44

Page 1 05 Oct 00 10:14

Typist: LLR

ATTACHMENT 2
Case Narrative Form (Data Package Format)

(Example Only)

Case Narrative Form (Data Package Format), Ref: SWL-OV-103

SOUTHWEST LABORATORY OF OKLAHOMA
1700 West Albany, Suite A
Broken Arrow, OK 74012
918-251-2858

SDG NARRATIVE

August 25, 1999

CLIENT:

SDG No.:

VOLATILE FRACTION

Seven soil samples and one water sample were submitted for Volatile Organic Analysis. The samples were analyzed by GC/MS following the SW846-8240 Statement of Work.

No major problems occurred during the analyses of these samples.

Blanks: No problems.

Surrogates: No problems.

Laboratory Control Spikes: LCS1, LCSD1, LCS2 and LCSD2 each contained one compound outside QC recovery limits. LCS3 and LCSD3 each contained two compounds outside QC recovery limits. No reanalyses were performed.

Internal Standards: No problems.

Harry M. Borg
Organic Program Manager

August 25, 1999

Typist: LLR

ATTACHMENT 3
Case Narrative Form (CLP Format)

Example Only

SOUTHWEST LABORATORY OF OKLAHOMA
1700 West Albany, Suite A / Broken Arrow, OK 74012
918-251-2858

SDG NARRATIVE
August 25, 1999

CONTRACT NO.:

CASE NO.:

SAMPLE NOS.:

SDG NO.:

VOLATILE FRACTION

Nineteen soil samples and one water sample were submitted for Volatile Organic Analysis. The samples were analyzed by GC/MS following the OLM03.2 CLP Statement of Work.

Alternate columns used for the analysis of volatile compounds by Method OLM03.2 are the Restek XTI-5 (bonded 5% phenyl-95% dimethyl polysiloxane), 30m, 0.25mm ID, 1um film thickness (Restek #12253) and the DB624, 75m, 0.53mmID Megabore, 3um film thickness (J&W 125-1374).

An alternate trap used for the analysis of volatile compounds by method OLM03.2 is the Vocarb 3000 (Carbopack B/Carboxen 1000 & 1001; Tekmar #2-1066).

The following samples in this SDG (labeled with an "RE") are considered billable since reanalysis was performed to verify internal standard area recoveries:

The following samples in this SDG (labeled with an "RE") are considered billable since reanalysis was performed to verify surrogate recoveries:.

The following samples in this SDG (labeled with a "DL") are considered billable since these samples were diluted to bring target analytes within the linear range:

No major problems occurred during the analyses of these samples.

Blanks: No problems.

Surrogates: No problems.

Matrix Spikes: No problems.

Internal Standards: No problems.

NOTE: All manual integrations in this data package for GC/MS Volatiles have been performed for one of the following reasons:

a. Data system missed peak during acquisition.
b. Data system improperly integrated peak.

If water samples are contained in this case, their pH data is included on the page accompanying this SDG narrative.

I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed above. Release of the data contained in this hardcopy data package and in the computer-readable data submitted on floppy diskette has been authorized by the Laboratory Manager, or his designee, as verified by the following signature.

Harry M. Borg
Organic Program Manager

August 25, 1999

SOUTHWEST LABORATORY OF OKLAHOMA, INC.

TM

Standard Operating Procedure

Volatile Data Clerks

Document No.: SWL-OV-106

Rev No. / Date: Rev. 1.0 — 09/01/98

OFFICIAL COPY RED STAMP INDICATES ORIGINAL

APPROVALS	DATES
Gina Jackson Procedure Prepared By	9/2/98 Date
Harry M-Long Organic Program Manager	9-8-98 Date
Chuck Jones Laboratory Safety Officer	9/8/98 Date
Chuck Jones Laboratory QA/QC Officer	9/8/98 Date

(Effective Date is 5 calendar days after the last signature above - QA/QC Officer)

Document Status

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VOLATILE DATA CLERKS

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1.0 SCOPE AND APPLICATION

1.1 The purpose of this standard operating procedure is to provide guidelines for data preparation performed by the Volatile Department Data Clerks. It will provide specific instructions related to the assembly and completion of standard tabular reports and data packages.

2.0 RESPONSIBILITIES

2.1 Volatile Sample Log-In

2.2 Episode Worksheet Distribution to Analysts

2.3 Removal of Completed Samples From LIMS

2.4 Calibrations

2.4.1 Copying

2.4.2 Filing

2.5 Data Package Assembly

2.6 Data Package Submittal for Initial Review

2.7 Processing of GC/MS Data Packages

2.8 Processing of GC Volatile Data Packages

2.9 Error Correction

2.10 Generation of Sample Holding Time Report

3.0 EQUIPMENT

3.1 PC: Gateway 2000: 486/100 or equivalent

3.2 LIMS

3.3 Enviroquant

3.4 Target 3.0 or Equivalent

3.5 Envision Forms Generation Software 3.0 or Equivalent

3.6 Ingres 6.3/02P

3.7 Unix A.09.01

3.8 MOSAIC Intranet

4.0 PRECAUTIONS—N/A

5.0 PROCEDURE

5.1 Sample Episode Log-In & Distribution

5.1.1 Pick up episode worksheets (See Attachment 1) from Sample & Receiving every morning from VOA tray outside Sample & Receiving. Samples must be logged in every morning, except Saturday and Sunday.

5.1.2 Print out the Department Test Status Report which is a list from LIMS of all samples received on previous day, or in the case of a weekend for Friday through Sunday (See Attachment 2). Compare to the episode worksheets received from Sample & Receiving, verifying receipt of all worksheets. Also, print Client/Project Status Reports (individual summary sheets of the episodes). Print these sheets in the following manner:

5.1.2.1 At the LIMS PC, log onto the LIMS system

5.1.2.2 Select "Worksheet Options".

5.1.2.3 Select "Department/Test Status" and fill in the following fields: *Start Date: 07/23/98, End Date: 07/23/98, Sample Status: A, Matrix: A, Report Type: D, Dept.: MS, Sort by: S.*

5.1.2.4 Select "Printer" by pressing Cntl-B simultaneously and highlighting "volab" on the list of printers: Hit F10 twice to change printer destination.

5.1.2.5 Print list of all samples received previous day by Hitting F10 twice

5.1.2.6 Escape out of the "Department/Test Status" page to "Worksheet Options" and select "Client/Project Status Report".

5.1.2.7 In "Client/Project Status Report", fill in the following fields: *Client:___, Start Date: 07/23/98, End Date: 07/23/98, Matrix:____, and Report Type: T, then fill in test code when prompted.* One report for each matrix must be printed. (See Attachments 3 & 4)

5.1.2.8 Print reports in same manner as other reports above.

5.1.3 Attach "Client/Project Status Reports" to episode worksheets and note the following items at the top of the page:

- CLP or Non-CLP (According to the Analytical Request Form [ARF] attached to the back of each episode. (See Attachment 5)
- Name of Analyst responsible for analyzing that episode.
- Holding Date (using date stamp)

- Turnaround Time (TAT)

If samples are Rapid (less than a 21 day TAT episode), stamp the bottom of the Client/Project Status Report with the green ink stamp informing them to submit the episode and its corresponding calibration directly to the Data Reviewer.

5.1.4 Enter episode(s) into "VOA SAMPLE LOG FORM" (See Attachment 6). Enter the Date Samples Received, Test Codes, Episode Number, Client, Project, Matrix, (Soils and Waters *must* be entered on separate lines.), Number of Samples, Analyst, TAT, and CLP vs. Non-CLP. The "Analysts' Initial" field will be filled in by the analyst when they accept ownership of assigned worksheets.

5.1.5 Prepare a Client Label which includes Client Name, Episode Number, Matrix(s) of Samples, Actual Date Due, SDG (Sample Delivery Group), and Test Code. Place label on shelf according to episode number.

5.1.6 Fill out calendar according to Due Date. Write Non-CLP Client and SDG number on the calendar 2 days before it's due. Add CLP cases to the calendar as Sample SDG Reports (See Attachments 7A &7B) are received from BNA's.

5.1.7 Distribute episode worksheets assigned to analysts and have each analyst sign the VOA Sample Log Form Book, verifying they received all episodes assigned to them.

5.2 Removing Completed Samples Off LIMS

5.2.1 When episodes are analyzed and returned to the Data Clerks by the Analysts, the samples need to be removed from the LIMS and logged back in the VOA Sample Log Form.

5.2.1.1 Locate the completed episode number in the VOA Sample Log Form and enter the date the Analyst returned the samples to the Data Clerks.

5.2.1.2 At the LIMS PC, log onto the LIMS.

5.2.1.3 Select "Results Management".

5.2.1.4 Select "Manual Results Entry by Test".

5.2.1.5 Enter the test code of the episode you wish to remove. The LIMS will find the oldest episode with that test code. You may accept that episode or key in the specific episode needed.

5.2.1.6 When the requested episode appears on the screen, enter the date analyzed, time analyzed, and Analysts' initials. Press F10 twice.

5.2.1.7 Place completed episode on shelf above specific client label.

5.3 Calibrations

5.3.1 After the Data Reviewer has checked an Analyst's calibration, it is brought to the Data Clerks for the following functions:

- Check calibration order
- Verify manual integration print-outs
- Verify Analysts' initials/dates
- Note on Calibration Log Form in the respective place using a "C" for Continuing Calibrations and "I" for Initial Calibrations. (See Attachment 8)
- Make all necessary copies
- File original calibration according to instrument and date
- Place calibration copies with episode in shelf

5.4 GC/MS Data Package Assembly

5.4.1 Non-CLP Data (Standard Tabular Reports) See Attachment 9.

5.4.1.1 Fill out VOA Case Status Form with all information from episode worksheet. See Attachment 10.

5.4.1.2 Print % Moisture Report using MOSAIC for Soil samples and verify any water samples in the episode/SDG have a pH Form (See Attachment 11) submitted by the analysts.

5.4.1.3 Check samples for correct Client ID and Episode Number.

5.4.1.4 Verify all associated calibration files are present.

5.4.1.5 Remove BFB's and calibration chromatograms & manual integrations, leaving only the continuing calibration quant report. Place the BFB and calibration information in Extra Data.

5.4.1.6 Number the method blanks (VBLK1, VBLK2, etc.) according to the date and time the method blanks were analyzed.

5.4.1.7 Number the LCS/LCSD's the same as the method blanks.

5.4.1.8 Bind the case, log into Completed Episodes Notebook, and give to the Data Reviewer for final review and delivery to the Reporting Department.

5.4.2 CLP-Like Data Packages (See Attachment 12)

5.4.1.1 Fill out VOA Case Status Form with all information from episode worksheet.

5.4.1.2 Print % Moisture Report using MOSAIC for Soil samples and verify any water samples in the episode/SDG have a pH form submitted by the analysts.

5.4.1.3 Check samples for correct Client ID and Episode Number.

5.4.1.4 Verify all associated calibration files are present, both initial and continuing.

5.4.1.5 Number the method blanks (VBLK1, VBLK2, etc.) according to the date and time the method blanks were analyzed.

5.4.1.6 Number the LCS/LCSD's the same as the method blanks.

5.4.1.7 Insert copies of all associated analyst run log pages.

5.4.1.8 Bind the case, log into Completed Episodes Notebook, and give to the Data Reviewer for initial review, prior to processing the forms package.

5.5 GC/MS Data Package Forms Generation

5.5.1 Refer to SWL-OV-200.

5.6 GC Volatiles Data Package Assembly

5.6.1 Non-CLP Data (Standard Tabular Reports)

5.6.1.1 Fill out Case Status Form with all information from episode worksheet.

5.6.1.2 Place analyst-generated Form 1's behind Client Cover Sheet.

5.6.1.3 Copy calibrations and place in Extra Data.

5.6.1.4 Verify all associated calibration files are present.

5.6.1.5 Bind the case, log into Completed Episodes Notebook, and give to the Data Reviewer for final review and delivery to the Reporting Department.

5.6.2 CLP-Like Data Packages

5.6.2.1 Fill out Case Status Form with all information from episode worksheet.

5.6.2.2 Make copies of all associated calibrations.

5.6.2.3 Generate forms as described below in Section 5.7

5.7 GC Volatile Forms Generation Using Enviroquant

5.7.1 At LIMS PC, double-click on GC VOA Forms. This will bring up the Forms Generation Software.

5.7.2 Click on "Case". Click on "New SDG".

5.7.3 Select Fraction.

5.7.4 Enter General Header Information:

- Case Number (Client Name)
- SDG Number

5.7.5 Import List. Select Compound List to Import.

5.7.6 Select Spike List to Import.

5.7.7 Select LCS List to Import.

5.7.8 Click on "Case". Click on "Set Default" and enter Default Information.

5.7.9 Click on "SDG". Click on "Import Data". If calibrations have been used before, skip to *5.4.9*.

5.7.10 Select "File Type".

5.7.11 Select Calibration to Import. (#2 is TPH, all others are under instrument)

5.7.12 Select "Date". You may have to remove Continuing Calibration from that day.

5.7.13 Click on "SDG".

5.7.14 Click on "Assign File".

5.7.15 Click on F: Drive.

5.7.16 Select Data, Instrument, and Date.

5.7.17 Select the files you need and click on "Add".

5.7.18 Click on "Process".

5.7.19 Edit analysis data as needed.

5.7.20 Answer "Parsing Compound Information" questions.

- Remove False Positives?
- Was Power Curve Used?

5.7.21 Click on "Forms".

5.7.22 Click on "Generate Forms". (All or Partial)

5.7.23 Click on specific forms for review.

5.7.24 Click on "Print Forms".

5.7.25 Select forms to be printed by double-clicking on specific forms or by highlighting forms and clicking on "Add".

5.7.26 Print Forms.

5.8 Error Correction

5.8.1 It is the responsibility of data clerks to correct errors in an acceptable manner; line out the error, initial, and date. All corrections must be made using Black ink ONLY. 'WHITE-OUT' AND OBLITERATIONS ARE NOT ALLOWED.

5.9 Generation of "Holding Time Report" (See Attachment 14)

5.9.2 Print out a list from LIMS of all volatile samples received and their respective holding times:

5.9.1.1 At the LIMS PC, log onto the LIMS system

5.9.1.2 Select "Worksheet Options".

5.9.1.3 Select "Department/Week Summary" and fill in the following fields: *Start Date:(Earliest Date of Samples Not Analyzed) End Date: (Today's Date), Sample Status: A, Matrix: A, Report Type: D, Dept.: MS, Sort by: S.*

5.9.1.4 Select "Printer" by pressing Cntl-B simultaneously and highlighting "volab" on the list of printers: Hit F10 twice to change printer destination.

5.9.1.5 Print holding time list by Hitting F10 twice

6.0 RECORDS. N/A

7.0 REFERENCES

7.1 SWL-OV-200

7.2 SWL-OV-101

7.3 SWL-OS-301

8.0 ATTACHMENTS

8.1 Attachment 1 "Episode Worksheet"

8.2 Attachment 2 "Department Test Status Report"

8.3 Attachment 3 "Completed Client Status Report (21 Day TAT)"

8.4 Attachment 4 "Completed Client Status Report (Rapid TAT)

8.5 Attachment 5 "Analytical Request Form"

8.6 Attachment 6 "VOA Sample Log Form"

8.7 Attachment 7A"Sample SDG Report"

8.8 Attachment 7B "Sample SDG Report"

8.9 Attachment 8 "Calibration Log Form"

8.10 Attachment 9 "Assembly of Non-CLP Case Checklist"

8.11 Attachment 10 "Case Status Form"

8.12 Attachment 11 "pH Form"

8.13 Attachment 12 "Assembly of CLP Case Checklist"

8.14 Attachment 13 "Holding Time Report"

9.0 DEFINITIONS

9.1 Terminology

9.1.1 SDG-Narrative ..... Portion of the data package that includes sample episode or SDG information, and descriptive documentation of any problems encountered during analysis or forms generation.

9.1.2 Extra Data ..... Data classified as non-reportable, but stored by the laboratory with the pertinent episode information for reference purposes.

9.1.3 Enviroquant ..... Software package used to process GC Volatiles data packages

9.1.4 MOSAIC ..... Inter-laboratory intranet

9.2 Acronyms

9.2.1 ARF ..... Analytical Request Form

9.2.2 EPA ..... United States Environmental Protection Agency

9.2.3 LCS ..... Laboratory Control Spike

9.2.4 LIMS ..... Laboratory Information Management System

9.2.5 MS ..... Matrix Spike

9.2.6 MSD ..... Matrix Spike Duplicate

9.2.7 SOP ..... Standard Operating Procedure

9.2.8 SWL ..... Southwest Laboratory of Oklahoma, Inc.

9.2.9 TAT ..... Turn-Around-Time

ATTACHMENT 1

Episode Worksheet

SOUTHWEST LABORATORY OF OKLAHOMA, INC.
1700 W. ALBANY SUITE C
BROKEN ARROW, OK 74012-1421

Date: 06/18/98
Episode: 34438
Client: ERM-NM
Project: ATA PIPELINE

PLE LOG-IN RECORD

SAMPLE #	DATE IN	DESCRIPTION	SDG	MA	NC	TEST	PRI	DUE	CONTAINER	DESCRIPTION	RESULTS	ANALYST	DATE/TIME
34438.01	06/18/98	DRUMM-PW-0698 Sampled: 06/17/98		W	2	MS399	S	07/01/98	AB	VOA NON STD			
34438.02	06/18/98	DRUML-PW-0698 Sampled: 06/17/98		W	2	MS399	S	07/01/98	AB	VOA NON STD			
34438.03	06/18/98	DRUMM-PW-0698 Sampled: 06/17/98		W	2	MS399	S	07/01/98	AB	VOA NON STD			

Total samples = 3

REVIEWED
BY JM
DATE 6/18/98

ATTACHMENT 2

Department Test Status Report

DEPARTMENT/TEST STATUS REPORT
09-10-1997 07:14:32
Page: 1

Status: ALL
Matrix: W - Water

Time frame: 09/09/97 to 09/09/97

SID #	CLIENT	DESCRIPTION	TYP	SDG	RECEIVED	EXTRACTED	ANALYZED	RESULT	LIMIT	BY	DUE DATE
Test: MS399 - VOA NON STD											
30901.01	ENSAFE	SMEGW00503		30849	09/09/97			VOA NON STD :			09/29/97
30901.02	ENSAFE	SMEDW00503		30849	09/09/97			VOA NON STD :			09/29/97
30901.03	ENSAFE	SMEEW00503		30849	09/09/97			VOA NON STD :			09/29/97
30901.04	ENSAFE	SMEFW00503		30849	09/09/97			VOA NON STD :			09/29/97
30901.05	ENSAFE	SMETW00503		30849	09/09/97			VOA NON STD :			09/29/97
30906.01	ITCORPTN	UT2271		30906	09/09/97			VOA NON STD :			09/22/97
30906.02	ITCORPTN	UT2260		30906	09/09/97			VOA NON STD :			09/22/97
30906.03	ITCORPTN	UT2259		30906	09/09/97			VOA NON STD :			09/22/97
30906.04	ITCORPTN	UT2273		30906	09/09/97			VOA NON STD :			09/22/97
30906.05	ITCORPTN	UT2261		30906	09/09/97			VOA NON STD :			09/22/97
30906.06	ITCORPTN	UT2265		30906	09/09/97			VOA NON STD :			09/22/97
30906.07	ITCORPTN	UT2265	MS	30906	09/09/97			VOA NON STD :			09/22/97
30906.08	ITCORPTN	UT2265	MSD	30906	09/09/97			VOA NON STD :			09/22/97
30906.10	ITCORPTN	UT2264		30906	09/09/97			VOA NON STD :			09/22/97
30906.11	ITCORPTN	UT2263		30906	09/09/97			VOA NON STD :			09/22/97
30906.12	ITCORPTN	UT2257		30906	09/09/97			VOA NON STD :			09/22/97
30906.13	ITCORPTN	UT2267		30906	09/09/97			VOA NON STD :			09/22/97
30906.14	ITCORPTN	UT2269		30906	09/09/97			VOA NON STD :			09/22/97
30906.15	ITCORPTN	UT2270		30906	09/09/97			VOA NON STD :			09/22/97
30906.16	ITCORPTN	UT2272		30906	09/09/97			VOA NON STD :			09/22/97
30906.17	ITCORPTN	UT2256		30906	09/09/97			VOA NON STD :			09/22/97
30906.18	ITCORPTN	UT2258		30906	09/09/97			VOA NON STD :			09/22/97
30906.19	ITCORPTN	UT2262		30906	09/09/97			VOA NON STD :			09/22/97

ATTACHMENT 3

Comleted Client Status Report (21 Day TAT)

NCLP

SERETHA
21 DAY
9/19/98

CLIENT/PROJECT STATUS REPORT
09/10/1997 09:06:15
Page: 1

Client: ENSAFE
Status: ALL
Test: MS399 - VOA BY GC/MS NONSTANDARD

Project: ZONE F PO4 REL 26
Time frame: 09/09/1997 to 09/09/1997

SID #	RECEIVED	DESCRIPTION	TYP	MATRIX	TEST	DESCRIPTION	DUE	RESULTS	BY	REPORTED	REPORT #
30901.01	09/09/97	SMEGW00503		W	MS399	VOA NON STD	09/29/97				
30901.02	09/09/97	SMEDW00503		W	MS399	VOA NON STD	09/29/97				
30901.03	09/09/97	SMEEW00503		W	MS399	VOA NON STD	09/29/97				
30901.04	09/09/97	SMEFW00503		W	MS399	VOA NON STD	09/29/97				
30901.05	09/09/97	SMETW00503		W	MS399	VOA NON STD	09/29/97				

Total samples = 5
Total tests = 5

ATTACHMENT 4

Completed Client Status Report (Rapid TAT)

BECKY
14 DAY
9/19/97

CLP

CLIENT/PROJECT STATUS REPORT
09/10/1997 09:06:20
Page: 1

Client: ITCORPTN
Status: ALL
Test: MS399 - VOA BY GC/MS NONSTANDARD

Project: TINKER AFB
Time frame: 09/09/1997 to 09/09/1997

SID #	RECEIVED	DESCRIPTION	TYP	MATRIX	TEST DESCRIPTION	DUE	RESULTS	BY	REPORTED	REPORT #
30906.01	09/09/97	UT2271		W	MS399 VOA NON STD	09/22/97				
30906.02	09/09/97	UT2260		W	MS399 VOA NON STD	09/22/97				
30906.03	09/09/97	UT2259		W	MS399 VOA NON STD	09/22/97				
30906.04	09/09/97	UT2273		W	MS399 VOA NON STD	09/22/97				
30906.05	09/09/97	UT2261		W	MS399 VOA NON STD	09/22/97				
30906.06	09/09/97	UT2265		W	MS399 VOA NON STD	09/22/97				
30906.07	09/09/97	UT2265	MS	W	MS399 VOA NON STD	09/22/97				
30906.08	09/09/97	UT2265	MSD	W	MS399 VOA NON STD	09/22/97				
30906.10	09/09/97	UT2264		W	MS399 VOA NON STD	09/22/97				
30906.11	09/09/97	UT2263		W	MS399 VOA NON STD	09/22/97				
30906.12	09/09/97	UT2257		W	MS399 VOA NON STD	09/22/97				
30906.13	09/09/97	UT2267		W	MS399 VOA NON STD	09/22/97				
30906.14	09/09/97	UT2269		W	MS399 VOA NON STD	09/22/97				
30906.15	09/09/97	UT2270		W	MS399 VOA NON STD	09/22/97				
30906.16	09/09/97	UT2272		W	MS399 VOA NON STD	09/22/97				
30906.17	09/09/97	UT2256		W	MS399 VOA NON STD	09/22/97				
30906.18	09/09/97	UT2258		W	MS399 VOA NON STD	09/22/97				
30906.19	09/09/97	UT2262		W	MS399 VOA NON STD	09/22/97				
30906.20	09/09/97	UT2266		W	MS399 VOA NON STD	09/22/97				

Total samples = 19
Total tests = 19

RAPID CASE
PLEASE GIVE CALIBRATIONS
AND RAW DATA TO JANET BY:
~~REQUIRES ADVANCE FORM 1'S~~

ATTACHMENT 5

Analytical Request Form (ARF)

PAGE 1

SOUTHWEST LABORATORY OF OKLAHOMA
ANALYTICAL REQUEST FORM

CLIENT: Environmental Resource Management
CONTACT: Chris Bynum
PROJECT: ATA PIPELINE
LOCATION: NEW MEXICO
DELIVERABLES: CLP DATA PACKAGE & FORM 1s
SDG:

CLIENT COD ERM-NM
PHONE NO: 505-243-3330
PO #
RCPT DATE: 06/18/98
SAMPLE TAT 14 DAYS TAT

CLIENT SAMPLE ID	SAMPLE MATRIX	(VOA) *MS399	(PNA) MS100	(EDB) GC160	(GRO) GC327	flashpt HW350						
DRUMM-PW-0698	W	X										
DRUML-PW-0698	W	X										
DRUMH-PW-0698	W	X										

SPECIAL PROVISIONS: PROPSAL # 4911

REPORTING ADDRESS * SEE ATTACHED LIST

BILLING ADDRESS

ML PROJECT OFFICER RANDY STAGGS

DATE PREPARED: 18-Jun-98 11:18
(VER1.0-7/5/94)

ERM04513.WB1

ATTACHMENT 6

VOA Sample Log Form

IN-HOUSE VOA SAMPLE LOG

VOLATILES GCMS DEPARTMENT

VRFY SMPLS RCVD	DATE SAMPLES RECEIVED	MS TEST CODES	EPISODE #	CLIENT	PROJECT	MATRIX S / W	# OF SPLS	ANALYST SPL ASSIGNED TO	ANALYST INITIALS	DATE SPLS RETURNED	MISC. INSERT TAT CIRCLE FORMAT
											NONCLP CLP
											NONCLP CLP
											NONCLP CLP
											NONCLP CLP
											NONCLP CLP
											NONCLP CLP
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											NONCLP CLP
											NONCLP CLP

SWL/ SWL/AATB 1700 W. ALBANY, BROKEN ARROW, OKLAHOMA 74012 OFFICE (918) 251-2858 FAX (918) 251-2599 (VGMD15-0394-02)

ATTACHMENT 7A

Sample SDG Report

SAMPLE DELIVERY GROUP (SDG)
TRAFFIC REPORT (TR) COVER SHEET

LAB NAME: SOUTHWEST LABORATORY OF OKLAHOMA CONTRACT NO.: 68-D5-0026

LAB CODE: SWOK CASE NO.: 26242 SAS NO.: _____

FULL SAMPLE ANALYSIS PRICE IN CONTRACT: $ 877.00

SDG No./First Sample in SDG: EBYQ1 Sample Receipt Date: 06/03/98 (MM/DD/YY)
(Lowest EPA Sample Number in first shipment of samples received under SDG).

Last Sample in SDG: EBYR0 Sample Receipt Date: 06/03/98 (MM/DD/YY)
(Highest EPA Sample Number in last shipment of samples received under SDG).

DUE 06/15 12

EPA Sample Numbers in the SDG (listed in alphanumeric order):

1)	EBYQ1		34229.01	W	V-B-P	11)		
2)	EBYQ2		.02	W	V-B-P	12)		
3)	EBYQ3		.03	W	V-B-P	13)		
4)	EBYQ4		.04	W	V-B-P	14)		
5)	EBYQ5	MS/MSD	.05	W	V-B-P	15)		
6)	EBYQ6		.06	W	V-B-P	16)		
7)	EBYQ7		.07	W	V-B-P	17)		
8)	EBYQ9	RIN	.08	W	V-B-P	18)		
9)	EBYR0	RIN	.09	W	V-B-P	19)		
10)						20)		

Note: There are a maximum of 20 field samples in a SDG.

Attach Traffic Reports to this form in alphanumeric order
(i.e., the order listed on this form).

Sample Custodian _____ Date _____

ATTACHMENT 7B

Sample SDG Report

SDG Report

Sample Delivery Group (SDG)

TRAFFIC REPORT (TR) COVER SHEET

LAB NAME: SOUTHWEST LABORATORY OF OKLAHOMA
LAB CODE: SWOK CLIENT: KAISHILL
PROJECT: ROCKY FLATS
SDG#34144 DATA PACKAGE
DUE 6-15 18
6-9 12 FORMS + NA/

Samples in SDG

	Client ID	Lab ID	Matrix	Received	Test Codes----------
1	98A2143-001.002	34144.01	WR	05/28/1998	MS399
2	98A2143-002.002	34144.02	WR	05/28/1998	MS399
3	98A2143-003.002	34144.03	WR	05/28/1998	MS399
4	98A2143-012.002	34144.04	WR	05/28/1998	MS399
5	98A2143-013.002	34144.05	WR	05/28/1998	MS399

BEKKI CLANCY
Sample Custodian

5-29
Date

ATTACHMENT 8

Calibration Log Form

VOLATILES GC/MS CALIBRATION LOG

VOLATILES GC/MS DEPARTMENT

For the Month of: _____ , 19

	INSTRUMENT																							
	C			I			K			L			N			R			U		
DAY	a.b	b.b	c.b	a.b	b.b	c.b	a.b	b.b	c.b	a.b	b.b	c.b	a.b	b.b	c.b	a.b	b.b	c.b	a.b	b.b	c.b
1																					
2																					
3																					
4																					
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28																					
29																					
30																					
31																					

ATTACHMENT 9

Assembly of Non-CLP Case Checklist

CHECK LIST FOR NON-CLP DATA PACKAGE

Private clients: Use all LCS/LCSD's run with samples; except MS318 only use the 1st LCS/LCSD.

_____ % Moisture Sheet (Soils Only) TCLP Extraction Sheet (MS340 only)

_____ pH Log Sheets (All waters except P.E. samples)

_____ Check Sample names against Worksheet

_____ Write DL's, RE's and MS/MSD's on SDG sheet for Janet.

_____ Separate samples into Instrument, Date, File ID order.

_____ Match Samples to Continuing Calibrations (add Sample changes to SIS sheet)

_____ Separate VBLK's, LCS and LCSD's. Number VBLKS's, LCS/D's

_____ Put Recovery Reports in front of VBLK, LCS/LCSD, IS reports in Extra Data

_____ Number VBLK's, LCS and LCSD's. Number by date and time of VBLK.

_____ Highlight the SIS (Sample Information Summary) all samples for that day.

_____ Rubber band each day for data reviewer

_____ Put Case in this order for Review

Review Order: Case Status Form, pH logs, Standards Log (MKE only)
Qualifier Page, TCLP Extraction Sheet (MS340 only), % Moisture Sheets,
Worksheets. Continuing Calibration/ Samples. Screen Data. Extra Data.

_____ Log Case in Gray Book, mark off calendar

_____ Give case to Data Reviewer.

ATTACHMENT 10

Case Status Form

VOA CASE STATUS FORM

Date :_____

CLP NONCLP

MS_____ # of Samples_____

Client/Case:_____

Data pkg. _____

FORMS ONLY_____

SDG/Episode:_____

Episode #'s:

_____

_____

Lab Code: SWOK / AATS

AIR

_____

_____

Case written up by:_____

ALL WATERS

_____

_____

pH Form:_____

ALL SOILS

% Moisture Sheets:_____

MIXED SAMPLES

Date Due:_____

TCLP Extraction Sheets:____

TAT_____ day

Clerks Notes:

_____

_____

_____

_____

_____

_____

_____

Data Reviewer Notes:

ATTACHMENT 11

pH Form

VOLATILES
GC/MS pH FORM
Ref: SWL-OV-101, Rev. 1.0
Form ID: OV-101-pH-F
Location/File ID: F:\COMMON\ORGANICS\PHLOG.DOC

CASE Knoxville SDG# _____

LAB ID	CLIENT SAMPLE ID	pH	ANALYST	DATE TESTED	DATE P.O. NOTIFIED
34484.01	98D220[illegible]	<2	[illegible]	10/29/98	
34484.05	98D2206-004MS	<2	↓		

COMMENTS: _____

* Date P.O. Notified: Field is filled out when samples are not acidified and Project Officer is notified of situation

ATTACHMENT 12

Assembly of a CLP Case Checklist

CHECK LIST FOR CLP DATA PACKAGE

Private clients: Use all LCS/LCSD's run with samples; except MS318 only use the 1st LCS/LCSD. MS317, 315 and 318 need storage/holding blanks.

_____ % Moisture Sheet (Soils Only) TCLP Extraction Sheet (MS340 only)

_____ pH Log Sheets (All waters except P.E. samples)

_____ Standards Log (Client MKE only)

_____ Check Sample names against Worksheet

_____ Write DL's, RE's and MS/MSD's on SDG sheet for Janet.

_____ Separate samples into Instrument, Date, File ID order.

_____ Match Samples to Continuing Calibrations (add Sample changes to SIS sheet)

_____ Separate VBLK's, LCS and LCSD's. Remove IS & Surrogate Sheets.
Number VBLK's, LCS and LCSD's. Number by date and time of VBLK.
Not by Instrument

_____ Highlight the SIS (Sample Information Summary) all samples for that day.

_____ Pull Initial Calibrations for Continuings

_____ Calibrations Stamped (EPA ONLY!!!)

_____ Pull Log Runs for each day samples,calibrations were run. Including Initials

_____ Circle SWOK or AATS on Run Logs. Put them into Instrument, Date order.

_____ Put case in this order to be reviewed. Rubber Band Case.

Review Order: Case Status Form/SDG, pH logs, Standards Log (MKE only)
Qualifier Page, TCLP Extraction Sheet (MS340 only), % Moisture Sheets,
Worksheets. Run Logs. Calibration/ Samples. Screen Data. Extra Data.

_____ Log Case in Gray Book, put red dot on calendar.

_____ Give case to Data Reviewer.

ATTACHMENT 13

Holding Time Report

SOUTHWEST LABORATORY OF OKLAHOMA, INC.
Department Week Report — Department : MASS SPECTROMETRY — Date : 09-10-1997 — Page : 2
Test : MS3XX — Report : Holding Times — Start date : 09-01-1997

Test code: MS399 VOA BY GC/MS NONSTANDARD

S: 10 days W: 10 days O: 10 days

Mon 09/01/97	Tues 09/02/97	Wed 09/03/97	Thur 09/04/97	Fri 09/05/97	Sat 09/06/97	Sun 09/07/97
W 30563.01 C /1	W 30577.01 C /1	W 30613.01 C /1	W 30638.01 C /1	W 30652.01 C /1	W *30819.06 C	WR 30742.01 C /1
W 30563.02 C /1	W 30577.02 C /1	W 30613.02 C /1	W 30638.02 C /1	W 30652.02 C /1	W *30819.08 C	WR 30742.02 C /1
W 30563.03 C /1	W 30577.03 C /1	W 30613.03 C /1	W 30638.03 C /1	S 30655.01 C /1		WR 30742.03 C /1
W 30563.07 C /1	W 30577.04 C /1	W 30613.04 C /1	W 30638.04 C /1	S 30655.02 C /1		WR 30742.05 C /1
W 30570.01 C /1	W 30577.05 C /1	W 30613.05 C /1	W 30638.05 C /1	S 30655.03 C /1		WR 30742.07 C /1
W 30570.02 C /1	S 30578.01 C /1	W 30613.06 C /1	W 30638.06 C /1	S 30655.04 C /1		WR 30742.09 C /1
W 30570.03 C /1	S 30578.02 C /1	W 30629.01 C /1	W 30638.07 C /1	S 30655.05 C /1		WR 30742.11 C /1
W 30570.04 C /1	W 30578.04 C /1	W 30629.02 C /1	W 30638.08 C /1	S 30655.06 C /1		W *30827.01 I
W 30570.05 C /1	S 30578.05 C /1	W 30701.01 C /1	W 30638.09 C /1	S 30655.07 C /1		
W 30570.06 C /1	S 30578.06 C /1	W 30701.02 C /1	W 30638.10 C /1	S 30655.08 C /1		
W 30570.07 C /1	S 30578.07 C /1	W 30701.03 C /1	W 30639.01 C /1	W 30655.09 C /1		
W 30571.02 C /1	S 30578.08 C /1	W 30701.04 C /1	W 30639.02 C /1	W 30726.01 C /1		
W 30571.03 C /1	S 30578.09 C /1	W 30701.05 C /1	W 30639.03 C /1	W 30726.02 C /1		
W 30571.04 C /1	S 30578.11 C /1	W 30701.06 C /1	W 30639.04 C /1			
W 30571.05 C /1	W 30598.01 C /1	W 30701.07 C /1	W 30639.05 C /1			
W 30671.01 C /1	W 30598.02 C /1	W 30701.08 C /1	W 30640.01 C /1			
W 30671.02 C /1	W 30598.03 C /1	W 30701.09 C /1	W 30640.02 C /1			
W 30671.03 C /1	W 30598.04 C /1	W 30701.10 C /1	W 30640.03 C /1			
W 30671.04 C /1	W 30598.05 C /1	W 30701.11 C /1	W 30640.04 C /1			
W 30671.05 C /1	W 30693.01 C /1	W 30701.12 C /1	S 30643.01 C /1			
W 30671.06 C /1	W 30693.02 C /1	W 30701.13 C /1	S 30643.02 C /1			
W 30671.07 C /1	W 30693.03 C /1	W 30701.14 C /1	S 30643.03 C /1			
W 30671.08 C /1	W 30693.04 C /1	W 30701.15 C /1	S 30643.04 C /1			
W 30671.09 C /1	W 30693.05 C /1	W 30701.16 C /1	S 30643.05 C /1			
W 30671.10 C /1	W 30693.06 C /1	W 30701.17 C /1	S 30643.06 C /1			
W 30671.11 C /1	W 30693.07 C /1	WR 30703.01 C /1	W 30711.01 C /1			
W 30671.12 C /1	W 30693.08 C /1	WR 30703.02 C /1	W 30711.02 C /1			
	W 30693.09 C /1	WR 30703.03 C /1	W 30711.03 C /1			
	W 30693.10 C /1	WR 30703.04 C /1	W 30711.05 C /1			
	W 30693.12 C /1	WR 30703.05 C /1	W 30711.06 C /1			
	W 30693.13 C /1	WR 30704.01 C /1	W 30711.07 C /1			
	W 30693.14 C /1	WR 30704.02 C /1	W 30711.08 C /1			
	W 30693.15 C /1	WR 30708.01 C /1	W 30711.09 C /1			
	W 30693.16 C /1	WR 30708.02 C /1	W 30711.10 C /1			
	W 30693.17 C /1	WR 30708.04 C /1	W 30711.11 C /1			
		WR 30708.06 C /1	W 30711.12 C /1			
		WR 30708.08 C /1	W 30711.13 C /1			
			W 30711.14 C /1			
			W 30711.15 C /1			
			W 30711.16 C /1			
			W 30711.17 C /1			
			W 30711.18 C /1			
			W 30711.19 C /1			
			WR 30717.01 C /1			
			WR 30717.02 C /1			
			WR 30717.03 C /1			
			WR 30717.04 C /1			
			WR 30717.05 C /1			
			WR 30717.06 C /1			
			WR 30717.08 C /1			

H= Hold
C= Complete
I= In Complete
R= Reported

SOUTHWEST LABORATORY OF OKLAHOMA, INC. *and*
AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.

TM

Standard Operating Procedure

Analysis of Volatile Organics by GC/MS
SW846 Method 8260B

Document No.: SWL-OV-303

Rev No. / Date: Rev. 5.0 — 10/10/00

OFFICIAL COPY RED STAMP INDICATES ORIGINAL

APPROVALS	DATES
Procedure Prepared By	Date 10/11/00
Program Manager or Section Supervisor	Date 10-24-00
Laboratory Safety Officer	Date 10/24/2000
Laboratory QA/QC Officer	Date 10/24/00

(Effective Date is 5 calendar days after the last signature above - QA/QC Officer)

Document Status

[ ] Controlled DC No. _____ Issued to: _____ Date: _____ QA: _____

[✓] Official Copy OC No.: 2431 Issued to: Sandra Karsonovich Date: 11/14/00 QA: _____

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ANALYSIS OF VOLATILE ORGANICS BY GC/MS (SW846 METHOD 8260B)

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APPENDIX

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Typist: LLR

1.0 SCOPE AND APPLICATION

1.1 Method 8260B, test code MS326, is a purge-and-trap, gas chromatograph/mass spectrometer procedure used to determine volatile organic compounds in many different types of matrices regardless of water content. It can be used to quantitate most volatile organic contaminants with boiling points less than 200°C that are insoluble or only slightly soluble in water. It can also be used for the quantitation and analysis of the more soluble organic compounds such as ethers, acetates, acrylates, and ketones, but with quantitation limits increased accordingly due to poor purging efficiency. In general, for most contaminants, the reporting limit for this method for any one compound is 5 µg/kg for soil/sediment samples 5 µg/L for ground water samples, and 0.5 mg/kg for wastes. Clearly, reporting limits will be higher for sample extracts or samples requiring dilution due to high concentrations of contaminants.

1.2 The reporting limits and compounds listed in Table 7 are normal limits and compounds analyzed by this method.

1.3 This procedure contains no method deviations.

2.0 METHOD SUMMARY

2.1 An inert gas, helium, is bubbled through either an aqueous sample or heated solid sample contained in a specifically designed purging chamber at ambient temperature. The purgeables are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the purgeables are trapped. After purging is completed, the sorbent column is heated and backflushed with the helium to desorb the purgeables onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the purgeables that are then detected with a mass spectrometer.

2.2 The amount of sample analyzed will vary depending upon the nature of the sample (aqueous or non-aqueous) and the desired quantitation limits of the analysis. For normal aqueous samples, a 5mL aliquot is used. For normal non-aqueous solid samples, 5.0 grams plus 5 mL filtered tap water will be analyzed. For non-aqueous samples, Method 5030B is the standard purge & trap method used and addressed throughout the SOP. If a client requests Method 5035, the criteria stated in Appendix A are employed.

2.3 For aqueous samples, an aliquot of the sample is diluted with filtered tap water when dilution is necessary. A 5 mL aliquot of the dilution is taken for purging. For non-aqueous samples, as little as 1.0 gram may be used for low-level diluted analyses. For medium-level analyses, 4.0 grams of sample is extracted in 10 mL methanol, and up to 100 uL of the extract may be used.

2.4 If the amount of a compound detected is outside the linear range of the instrument, then a smaller sample size is analyzed.

2.5 All samples are analyzed within 14 days of the sampling date. (See Section 4.1 for details concerning sample preservation.)

3.0 HEALTH AND SAFETY

Some of the reagents solvents, and other chemicals used in this method have been clearly defined as toxic and/or carcinogenic; most, however, remain in question or are not precisely defined with respect to the hazards of handling. All chemicals and samples, therefore, are treated as potentially hazardous, and exposure to them is minimized. Also, the MSDS for all stock solutions, reagents, and solvents used are kept on file in an easily accessible place in the lab. Any spills that may occur are cleaned up immediately.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING & STORAGE

4.1 All water samples are to be preserved in the field using HCl. Verification of this preservation is performed by checking the pH of all water samples and recording their pH on the Volatile GC/MS pH Form. (Figure 1)

4.2 Samples are stored at 4° +/- 2° C. Standards are stored at –10° to –20°C. All samples and standard solutions must be allowed to warm to ambient temperature before analysis.

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

Many major sources of contamination are avoided by using stainless steel, nickel, polytetrafluroethylene (PFTE), glass, and other inert materials for all equipment and materials that in any way affect the detection and quantitation of volatile contaminants in samples. However, other areas of contamination do exist, and are avoided as follows:

5.1 Samples can sometimes be contaminated by diffusion of volatile organics through the septum seal during shipment and storage; this can be handled by the use of a trip blank prepared from organic-free filtered tap water which is carried through the entire sampling and shipping process with all samples, in addition to holding blanks prepared in the same fashion and stored in all areas where samples will be stored in the laboratory. Our laboratory encourages the analysis of a trip blank and sends trip blanks to clients when providing a bottle order for sampling.

5.2 Interfering contamination can occur when a sample is analyzed immediately following a sample containing high concentrations of volatile organic compounds. All such samples are reviewed carefully for any indication of carryover and reanalyzed when there is any question of whether contaminants found in the sample are the result of true contamination or originate from the previous analysis.

5.3 Common laboratory solvents present in the air of most analytical laboratories, notably methylene chloride, acetone, and chloroform, can cause contamination of the analytical instruments themselves; this can be checked by analyzing an instrument blank, where the presence of volatile contaminants can alert the analyst of the need to perform any cleaning measures necessary to achieve an analytical run which is as contaminant-free as possible.

6.0 APPARATUS AND MATERIALS

6.1 Micro syringes — 10 µL and larger, 0.006 inch ID needle.

Typist: LLR

6.2 Syringe valve — two-way, with Luer ends (three each), if applicable to the purging device.

6.3 Syringe — 5 mL, gas tight.

6.4 pH Paper — Range of 1-6

6.5 Balance, Analytical — capable of accurately weighing 0.0001 g. and a top-loading balance capable of weighing 0.1 g.

6.6 Glassware

6.6.1 Bottle — 40 mL, screw cap, with Teflon cap liner.

6.6.2 Volumetric flasks — class A with ground-glass stoppers.

6.6.3 Vials — 20mL for medium level extraction

6.7 Purge and trap device — The purge and trap device consists of three separate pieces of equipment; the sample purger, trap and the desorber. Heating blankets are added to the Tekmar 2032 and 2016 Autosamplers when low level soils are analyzed. Sample purgers include Tekmar ALS2032, ALS2016, Precept or Archon, or equivalent. Trap and desorber include Tekmar 2000 and 3000 or equivalent.

6.8 Gas chromatograph — An analytical system complete with a temperature programmable gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical column, and gases. GC is Hewlett-Packard 5890 Series II or equivalent.

6.9 Column — DB624, 75m, 0.53mm ID, 3.0 um (J&W Scientific Cat. No. 125-1334).

6.10 Jet Separator — Glass (SIS Cat. No. 113505).

6.11 Mass spectrometer — Capable of scanning from 35 to 300 amu every one second, utilizing seventy volts (nominal) electron energy in the electron impact ionization mode and producing a mass spectrum which meets all the criteria when 50 ng of 4-Bromofluorobenzene (BFB) is injected through the gas chromatograph inlet (see Table 3). MSD is Hewlett-Packard 5970, 5971, 5972, or equivalent.

6.12 Calibration requirements can be found in Table 6.

7.0 REAGENTS AND STANDARDS

7.1 Reagent water — Produced by filtering tap water through an organic filtration system containing activated charcoal prior to use.

7.2 Methanol — Pesticide quality or equivalent.

7.3 Stock standard solutions — Stock standard solutions are purchased and are traceable to EPA supplied standards. These solutions are stored in Teflon sealed screwcap bottles at -10°C to -20°C.

7.4 Secondary dilution standards — Using commercially prepared standard solutions, secondary dilution standards that contain the compounds of interest, either singly or mixed together, are prepared in methanol. Secondary dilution standards should be stored with minimal headspace and should be checked frequently for signs of degradation or evaporation especially just prior to preparing calibration standards from them. Secondary dilution standards are used until degradation is verified through daily calibration checks.

7.5 Internal Standard and Surrogate Spiking Solution — The internal standards are Pentafluorobenzene, 1,4-Difluorobenzene, Chlorobenzene-d_5, and 1,4-Dichlorobenzene-d_4. The surrogates are Dibromofluoromethane, Toluene-d_8, 4-Bromofluorobenzene, and 1,2-Dichloroethane d_4. Separate internal standard and surrogate standard solutions are prepared containing the above at a concentration of 25 µg/mL in methanol.

8.0 PROCEDURE

8.1 This method requires the following instrumental parameters:

8.1.1 Electron Energy: 70 Volts (nominal)

8.1.2 Mass Range: 35-300

8.1.3 Scan Time: to give at least 5 scans per peak, not to exceed one second per scan

8.2 Recommended Operating conditions for the gas chromatograph and column conditions: DB-624, 75m, 0.53mm ID, 3 µm film thickness at a flow rate of 30 mL/min. Column temperature is isothermal at 45°C for five minutes, then programmed at 5°C per minute to 105°C. further programmed at 10°C/min. to 180°C.

8.3 Adjust the purge gas (helium) flow rate to 35 ± 3 mL/min on the purge and trap. Variations from this flow rate may be necessary to achieve better purging and collection efficiencies for some compounds, particularly chloromethane and bromoform.

8.4 Tune

8.4.1 Before analysis of samples can begin, the instrument must meet all calibration criteria. The first step in the calibration of a Volatiles GC/MS system is to demonstrate the ionization and fragmentation of standard mass spectral tuning compounds. This, as well as a sensitivity check, is accomplished with the use of 4-Bromofluorobenzene, an EPA specified compound. This standard is run daily to validate the GC/MS system and to begin the 12-hour clock. BFB is analyzed by direct injection of a 50-ng aliquot into the instrument.

8.4.2 When the analysis is complete, the tune is checked against standard criteria (See Table 3 for Tune Criteria). SW8260 tune criteria for BFB must be met before any other calibration standards or sample analysis is performed.

8.4.3 If the tune fails to meet the criteria, BFB is rerun. If it fails again, the instrument must be retuned through "Auto Tune" or "Manual Tune," then BFB should be run again. If all criteria are met, the calibration sequence is run, but if it fails again, a supervisor is contacted because instrument maintenance such as source cleaning may be required.

Typist: LLR

8.5 Initial Calibration

8.5.1 Once BFB has met its tune criteria, calibration may begin. Initial calibration of the GC/MS is established and validated by the analysis of EPA traceable standards at five concentration levels which are 5, 20, 50, 100,and 200 ppb for the 5 mL or 5 gram purge method. These standards are purged on the Tekmar or Varian before analysis by GC/MS. The internal standard calibration procedure is used. In addition to surrogate recovery compounds, standards and sample extracts are spiked with internal standards that span the retention time range of the analytes of interest.

8.5.2 The Response Factors (RF) for the analytes in the standard are calculated and stored in a Target Method File. Using the RFs from the initial calibration, the percent relative standard deviation (%RSD) for all compounds is calculated utilizing the formula shown in Table 9. The criterion the initial calibration must meet to be acceptable is found in Table 6.

8.5.3 If the five-point does not meet criteria, each standard is checked for the need of a possible rerun, in case the standards have been added at the wrong amount, or the purge may have been bad. If reanalyzing a standard will not solve the problem, a supervisor is contacted. Some problems could be found to be related to purge chambers, multiplier, flows, etc.

8.5.4 If the initial calibration does meet the criteria stated in Table 6, and there is time remaining in the 12-hour time period, samples may be run.

8.5.4.1 If the % RSD of any compound is 15% or less, then the relative response factor is assumed to be constant over the calibration range, and the average relative response factor may be used for quantitation.

8.5.4.2 If the % RSD of any compound is greater than 15%, a calibration curve must be constructed of area ratios (A/Ais) versus concentration using first order regression fit of the five calibration points (ie: linear).

The use of calibration curves is a recommended alternative to average response factor calibration and a useful diagnostic of standard preparation accuracy and absorption activity in the chromatographic system.

A second-source, verification standard will be analyzed with each initial calibration. The % Difference from the initial calibration should be less than 50% for each compound.

8.5.5 The calibration standards are prepared by filling a syringe with filtered tap water and spiking with the standards as follows.

Soil - 5 mL Purge/Tekmar ALS2032/ALS2016

Internal Standard (µL)	Surrogate (µL)	25ppm Stock Std. (µL)	8260 Extra Cmpd.Stock Standard (µL)	Gases Stock Std. (µL)	Int. Std. Final Conc. (ng/mL)	Standard Final Conc. (ng/mL)
10	1	1	1	1	50	5
10	4	4	4	4	50	20
10	10	10	10	10	50	50
10	20	20	20	20	50	100
10	40	40	40	40	50	200

Soil-5mL Purge/Precept or Archon

Surrogate (µL)	25ppm Stock Std. (µL)	Gases Stock Std. (µL)	8260 Extra Cmpd. Stocks Standard (µL)	Int. Std. Final Conc. (ng/mL)	Standard Final Conc. (ng/mL)
8	8	8	8	50	5
32	32	32	32	50	20
80	80	80	80	50	50
160	160	160	160	50	100
320	320	320	320	50	200

8.5.6 If analyzing on the Tekmar 2032/2016, the syringe is attached to the syringe valve assembly on the Tekmar. The syringe valve is opened, the sample is injected into the purging chamber, and the purging device valve is closed. Water samples are purged at ambient temperature. Low-level soils are purged at 40° +/- 1°C. If analyzing on the Precept or Archon, the standard's 40mL volatile vial is loaded into the tray. The autosampler will pick up the vial, and do one of the two things: 1) If the vial is a soil standard, it will transfer the vial from the tray to a heated vial holder, spike it with internal standard, and purge the standard. 2) If the vial is a water standard, it will withdraw 5mL from the vial, spike it with internal standard, and transfer the 5ml to the Tekmar 3000 for purging. The sample is purged for 11.0 min at ambient temperature. At the conclusion of the purge time, the trapped materials are introduced to the GC column by the Desorb Mode where the trap is rapidly heated to a temperature specific to the trap being used.

8.5.7 After the sample desorbs onto the GC, the trap is baked for a minimum four minutes at a minimum of 225°C, then it is allowed to cool to less than 35°C before being ready to purge the next standard or sample. During this time, the Precept or Archon flushes its lines and needle.

8.5.8 The standard will be quantitated into a report in which concentrations, areas, response factors, standard deviations, percent differences, and regression coefficients are calculated.

Typist: LLR

8.6 Calibration Verification

8.6.1 A calibration verification of the GC/MS uses the same EPA traceable standards, but at only one level: 50 ppb. The analysis is performed in the same manner as stated before in the initial calibration. The calibration verification checks the same compounds as the initial calibration. A % Difference Report is produced for the CCC compounds and all other compounds using an average RRF. A % Drift Report is produced for those compounds using linear regression. The criteria the calibration verification must meet to be acceptable are also found in Table 6.

All other compounds should have a %Difference/Drift less than 50%, and must have a %Difference/Drift less than 100%. If the % Difference/Drift is greater than 100%, corrective action must be immediately performed, which includes reviewing the standard and contacting the supervisor. In addition, a comparison will be performed between the internal standard areas/retention times in the calibration verification and the initial calibration.

8.6.2 The calibration verification is performed after the BFB and must meet all criteria before any other analysis continues. If the CCC/SPCC criteria are not met, the standard must be rerun, as it is possible, the standard was not prepared correctly or that there was a bad purge. However, if the second standard still doesn't meet CCC/SPCC criteria, an initial calibration must be run or corrective action performed.

8.6.3 If the calibration verification does meet the criteria stated in Table 6, samples may be run. Samples are quantitated based on the criteria stated above in Section 8.5.4.

8.6.4 An additional standard will be run periodically (at least monthly) with the continuing calibration. This standard will be varied from the normal concentration used for the continuing calibration. Any of the other points can be used and the standard will be required to meet the same criteria as the continuing calibration.

8.7 Water Samples

8.7.1 Any headspace in the bottle is noted before opening. If analyzing on the Tekmar 2032 or 2016, the sample bottle (which has been allowed to come to ambient temperature) is opened and is carefully poured into the syringe barrel to just short of overflowing, then adjusted to a volume of 5 mL. This process of taking an aliquot destroys the validity of the sample for future analysis. Care must also be taken to prevent air from leaking into the syringe. The entire sample vial is loaded onto the Tekmar Precept or Varian Archon.

8.7.2 When spiking samples on the Tekmar 2032 or 2016, 10 µL of the Surrogate Spiking Solution and 10 µL of the Internal Standard Spiking Solution are added through the valve bore of the syringe, which is equivalent to a concentration of 50 µg/L of each standard. If samples are analyzed on the Tekmar Precept or Varian Archon, the instrument automatically spikes Internal Standard and Surrogate during analysis.

8.7.3 Water samples are loaded in the following manner to ensure the proper sample is placed into its designated purge chamber on the Tekmar 2032/2016 Autosampler:

- A test tube rack is filled with the samples' 40 mL VOA vials. The syringes to be used are placed in front of the samples.
- All sample purge tubes are labeled with the SWLO ID Number and Autosampler position. Each analyst verifies as he/she loads each sample into the purge chambers that the syringe matches the ID on the purge tube.
- The Tekmar Precept or Varian Archon allows the analyst to load the sample vials directly on the instrument, reducing the risk of a loading error. Purge in the same manner as stated during calibration.

8.7.4 For water samples analyzed on the Tekmar 2032 or 2016 requiring matrix spike and matrix spike duplicates, 10 µL of the matrix spike solution is added to the sample to be purged. Disregarding any dilutions, this is equivalent to a concentration of 50 µg/L of each matrix spike standard. Water samples analyzed on the Precept or Archon requiring MS/MSD, 80µL of the matrix spike solution is added to the sample to be purged, yielding a concentration of 50ug/L.

8.7.5 With all matrices, all dilutions should keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve.

8.7.6 An estimated concentration for Tentatively Identified Compounds (TICs) shall be quantified by the internal standard method. For quantification, the nearest internal standard shall be used.

8.7.7 The formula for calculating concentrations for target compounds is found in Table 9. For TICs, area counts from the total ion chromatograms are to be used for both the compound to be measured and the internal standard. A response factor (RF) of one (1) is to be assumed. The value from this quantitation shall be qualified as "estimated." This estimated concentration should be calculated for all TICs as well as those identified as unknowns.

8.7.8 After analysis is complete, the pH is recorded for all water samples on a pH Log (See Figure 1). If a sample has a pH reading >2, the project officer associated with that particular client's samples is notified.

8.8 Soil Sample Analysis - Low Level

8.8.1 The low-level method is based on purging a heated sediment/soil mixed with filtered tap water containing the surrogate and internal standards.

8.8.2 The sample (for volatile organics) consists of an equal representation of the entire contents of the sample container (i.e., no supernatant liquids are discarded.) The contents of the sample container are mixed with a narrow spatula, then the sample is weighed into a disposable test tube if analyzing on the Tekmar 2032 or 2016, or the sample is weighed into a disposable 40mL volatile vial if analyzing on the Tekmar Precept or Varian Archon. 5 grams of sample are used, unless a dilution is required, in which case 1 - 4 grams of sample may be used. A top loading balance is used, and the actual weight is recorded to the nearest 0.1 gram.

Typist: LLR

8.8.3 Immediately after weighing the sample into the labeled test tube or vial it is loaded onto the Tekmar ALS. If analyzing on the Tekmar 2032/2016, the plunger from a 5 mL Luerlock syringe is removed and the syringe is filled to overflowing with filtered tap water. The plunger is replaced to vent trapped air, then the volume is adjusted to 5.0 mL. 10 µL each of the Surrogate Spiking Solution and the Internal Standard Solution are added to the syringe. This addition to the soil is equivalent of 50 µg/Kg of each standard. This spiked filtered tap water is added to the test tube, the sample is heated to 40°C, and purging is begun. If analyzing on the Tekmar Precept or Varian Archon, the vial is transferred for heating and it automatically spikes the soil/water mixture with Internal Standard and Surrogate to yield a concentration for both of 50ug/Kg. The samples follow the same purge method as stated in the calibration.

8.8.4 For soil samples requiring matrix spike and matrix spike duplicates, 10 µL of the matrix spike solution is introduced into the 5 mL of the filtered tap water being added to the soil if analyzing on the Tekmar 2032/2016, or 80µL of matrix spike solution is introduced into 40mL of water if analyzing on the Tekmar Precept or Varian Archon. Disregarding any dilutions, this is equivalent to 50 µg/Kg of each matrix spike standard.

8.8.5 With all matrices, all dilutions should keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve.

8.8.6 An estimated concentration for Tentatively Identified Compounds (TICs) shall be quantified by the internal standard method. For quantification, the nearest internal standard shall be used.

8.8.7 The formula for calculating concentrations for target compounds is found in Table 9. For non-TIC compounds, area counts from the total ion chromatograms are to be used for both the compound to be measured and the internal standard. A response factor (RF) of one (1) is to be assumed. The value from this quantitation shall be qualified as "estimated." This estimated concentration should be calculated for all TICs as well as those identified as unknowns.

8.8.8 All soil samples must have their percent moisture determined so that compound contamination can be reported for "dry weight." This is performed by the Extractions Department and submitted to the Volatiles Data Clerk for reporting.

8.9 Soil Sample Analysis — Medium Level

8.9.1 Medium level extraction/analysis is required if analysis of a sample at a 1 gram level produces peak saturation, or compounds out of linear range. The medium level method is based on extracting the sediment soil sample with methanol. An aliquot of the methanol extract is added to filtered tap water containing the internal standards. The surrogate is spiked directly into the extract, thus making the addition directly into the filtered tap water unnecessary. The extract is purged at ambient temperature.

8.9.2 The medium level extract is prepared by weighing 4 grams of the sample into the scintillation vial. Using a top-loading balance, the actual weight is noted to the

- nearest 0.1 g, then 9900 µL of methanol and 100 µL of the medium concentration Surrogate Standard are added. The mixture is then capped and shaken by hand for 2 minutes. The extract should be made up quickly in order to avoid the loss of volatile organics.

8.9.3 The sample is loaded in the following manner: if analyzing on the Tekmar 2032/2016, a syringe is filled just short of overflowing using filtered tap water, then the volume is adjusted to 4.9 mL. The plunger is then adjusted to 5 mL to allow for the addition of the extract and standards, and 10 µL of Internal Standard Solution is added. Finally, the volume of the methanol extract and the volume of methanol, if needed, is added to total 100 µL. If analyzing on the Tekmar Precept or Varian Archon, a volatile vial is filled to 40mL and adjusted to 39.2mL. The volume of methanol extract and volume of methanol, if needed, is added to total 800µL. The methanol lot number is documented on the Encore Sample Preservation Form (Attachment 3).

8.9.4 The sample is loaded into the purging device (Tekmar) and the same purging method as stated during calibration is followed.

8.9.5 For samples requiring matrix spike and matrix spike duplicates, an extract is prepared in the same manner as stated above in Section 8.9.2, using 9800 µL of methanol, 100 µL of medium concentration surrogate, and 100 µL of medium concentration matrix spike solution. These samples are loaded in the same manner as other samples.

8.9.6 The method blank must be analyzed in the same manner as with low-level soils and waters, but it is prepared differently using 4 gram of a purified solid matrix. Surrogate Spiking Solution and methanol are then added to make a total volume of 10 mL, and the blank is purged at ambient temperature. An acceptable blank must contain less than or equal to the reporting limit of any volatile target compound, or else corrective action will be taken immediately.

8.9.7 With all matrices, all dilutions should keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve.

8.9.8 An estimated concentration for Tentatively Identified Compounds (TICs) shall be quantified by the internal standard method. For quantification, the nearest internal standard shall be used.

8.9.9 The formula for calculating concentrations for target compounds is found in Table 9. For TIC compounds, area counts from the total ion chromatograms are to be used for both the compound to be measured and the internal standard. A response factor (RF) of one (1) is to be assumed. The value from this quantitation shall be qualified as "estimated." This estimated concentration should be calculated for all TICs as well as those identified as unknowns.

8.9.10 All soil samples must have their percent moisture determined so that compound contamination can be reported for "dry weight." This is performed by the Extractions Department and submitted to the Volatiles Data Clerk for reporting. The values may also be obtained from the LIMs database by episode.

Typist: LLR

8.10 Analysis Report Criteria--A sample is acceptable when it meets the following criteria:

8.10.1 The internal standards' EICP areas are within a factor of 2(-50%/+100%) of the calibration' internal standards.

8.10.2 The internal standards' retention times are within ±30 seconds of the calibration's internal standards.

8.10.3 Surrogate recoveries are within the limits stated in Table 4.

8.10.4 Any compound found in the sample is within the linearity range, with any dilutions showing compounds in an acceptable range of linearity. If a sample analysis contains any target compounds above the linear range, all subsequent samples will be reviewed for those target compounds. The target compounds must be below the reporting limits to be acceptable; otherwise they must be reanalyzed.

8.10.5 Chromatogram shows no signs of carryover or instrument deficiencies.

8.11 Reanalysis Criteria: If sample does not meet internal standards' or surrogates' criteria, it must be reanalyzed. If the reanalysis meets all criteria, it is submitted. If the reanalysis duplicates the results from the first analysis, both analyses are submitted. This information is recorded in the analyst's log book.

8.12 A sample report is complete when it contains all of the following in order:

8.12.1 Chromatogram

8.12.2 Quant Report

8.12.3 Manual Integrations

8.12.4 Spectra of any compound, excluding internal standards and surrogates

8.12.5 Tentatively Identified Compounds (TICs)

8.12.6 Internal Standards/Surrogates

9.0 CALCULATIONS

All calculations are shown in Table 9 of this SOP.

10.0. QUALITY ASSURANCE/QUALITY CONTROL

10.1 Method Blank (Laboratory Reagent Blank)

10.1.1 The method blank is analyzed once the calibration has been met, whether it be an initial calibration or calibration verification. The method blank, called the "VBLK", consists of loading a syringe with 5mL of filtered tap water spiked to yield a concentration of 50 ppb internal standard and surrogate, or loading a 40mL volatile vial of filtered tap water into the Precept or Archon tray to be spiked automatically by the instrument. The VBLK is used to establish the analytical system is free of contaminants. It is also used to check for carryover

– contamination after a standard run.

10.1.2 An acceptable blank must meet the internal standard criteria stated in Section 8.10.1 and the surrogate recoveries found in Table 4. The concentration of the target compounds in the blank must be less than the method reporting limit for each target compound (see Table 7).

10.1.3 If a blank exceeds these criteria, corrective measures must be taken immediately, beginning with immediately reanalyzing the blank. If all criteria are still not met, the supervisor is then consulted.

10.2 Laboratory Control Spike (LCS and LCSD)

10.2.1 The Laboratory Control Spike (LCS and LCSD) are internal laboratory QC samples designed to show the capability of our laboratory to be in compliance with the protocol. They consist of filtered tap water spiked with an LCS solution which contains all compounds found in the daily calibration standards at a 50ppb level.

10.2.2 An acceptable LCS and LCSD must meet the internal standard criteria stated in Section 8.10.1 and the surrogate recoveries in Table 4. The percent recovery for each of the compounds in the LCS and LCSD should be within recovery limits shown in the QC Method Reference "Blue Book". The LCS limits are set forth by our laboratory showing our internal control limits, therefore, operators should put forth every effort to meet these limits. Limits are derived using a control chart program as per the Control Chart SOP (SWL-GA-112).

10.2.3 If the compounds are outside QC limits, corrective action is needed immediately, beginning with rerunning the LCS to verify that it was prepared correctly and that it was not a bad purge. If reanalysis does not solve the problem, a new standard may need to be made.

10.3 Matrix Spikes - MS/MSDs will be analyzed at a rate of 1 MS/MSD per batch of 20 or fewer samples. A minimum of the five compounds (1,1-Dichloroethene, Benzene, Trichloroethene, Toluene, and Chlorobenzene) will be used; however all compounds can be spiked. The recovery limits will be the same as the LCS/LCSD and will be advisory since they are dependent upon the matrix.

11.0 WASTE DISPOSAL/POLLUTION PREVENTION

Residual sample material and standards are discarded as directed by the Hazardous Waste Management Plan (SWL-GA-114.)

12.0 METHOD PERFORMANCE

12.1 The recovery limits for each of the compounds in the LCS and LCSD are shown in the QC Method Reference "Blue Book".

12.2 The recovery limits for each of the compounds in the MS and MSD are shown in the QC Method Reference "Blue Book".

12.3 MDL'S for this method are reported in the Laboratory's *"Red Book"*.

Typist LLR

12.4 Method 8260B is the analysis method used for several additional test codes. These test codes include TCLP Analysis, Skinner's List, Appendix IX, and BTEX. These specific test codes, sublists, and reporting limits for each of the above tests are shown in Tables 10-13.

13.0 REFERENCES

13.1 Method SW846-8260B (Update III, Revision 4 – December. 1996)

13.2 Method SW846-5030B (Update III, Revision 4, 1996)

13.3 Method SW846-5035 (Update III, Rev. 0, Dec. 1996)

13.4 Method SW846-5030A (Update I, July 1992)

13.5 Control Chart SOP (SWL-GA-112)

13.6 Definition and Determination of Detection Limits SOP (SWL-GA-113)

13.7 Hazardous Waste Management Plan SOP (SWL-GA-114)

14.0 DEFINITIONS

BFB:.....Bromofluorobenzene

Calibration: ..Establishment of an analytical curve based on response factors of known standards.

Calibration Standards:Series of known standard solutions used by the analyst for calibration of the instrument (i.e.: preparation of the analytical curve).

GC/MS:.....Gas Chromatography/Mass Spectrometry

LCS:.....Laboratory Control Spike: A known matrix spiked with compounds representative of the target analytes. Used to document laboratory performance.

15.0 DATA ARCHIVAL

Instrument data is stored in two separate locations before being archived to ensure data does not become erased or corrupt. Data is stored on both the instrument's Chemstation and the Windows NT File Server. An incremental backup of the server is performed daily, and a full backup of the server is performed weekly. The data is stored on the Chemstation for a minimum of 2 weeks.

16.0 METHOD DETECTION LIMITS (MDL'S)

16.1 MDL's are analyzed annually for both 5mL water and both soil methods (Method 5030 and Method 5035). Seven aliquots are analyzed at a concentration of 5ppb and their data is recorded in a spreadsheet format for easy review.

16.2 MDL's are verified to be below our reporting limit.

16.3 MDL'S are reported in the Laboratory's *"Red Book"*.

Table 1
Characteristic Ions for Surrogates and Internal Standards for Volatile Organic Compounds

Surrogate	Primary Characteristic Ion	Secondary Characteristic Ion(s)	Internal Standard	Primary Characteristic Ion	Secondary Characteristic Ion(s)
4-Bromofluorobenzene	95	174.176	Pentafluorobenzene	168	
Dibromofluoromethane	113		1,4-Difluorobenzene	114	
Toluene-d_8	98		Chlorobenzene-d_5	117	
1,2-Dichloroethane-d_4	65		1,4-Dichlorobenzene-d_4	152	

TABLE 2
Characteristic Masses (m/z) for Purgeable Organic Compounds

Analyte	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Acetone	43	58
Acetonitrile	41	40,39
Acrolein	56	55
Acrylonitrile	53	52, 51
Allyl Chloride	76	41,39
Benzene	78	—
Bromobenzene	156	77, 158
Bromochloromethane	128	49, 130
Bromodichloromethane	83	85, 127
Bromoform	173	175, 254
Bromomethane	94	96
2-Butanone	43	72, 57
n-Butylbenzene	91	92, 134
sec-Butylbenzene	105	134
tert-Butylbenzene	119	91, 134
Carbon Disulfide	76	78
Carbon Tetrachloride	117	119
Chlorobenzene	112	77, 114
[illegible]roethane	64	66
[illegible]roethylvinyl Ether	63	43, 43
[illegible]form	83	85
1-Chlorohexane	55	91,41
Chloromethane	50	52
Chloroprene	53	88
2-Chlorotoluene	91	126
4-Chlorotoluene	91	126
Cyclohexanone	55	42
cis-1,3-Dichloropropene	75	77
1,2-Dibromo-3-Chloropropane	75	155, 157
Dibromochloromethane	129	127
1,2-Dibromoethane	107	109, 188
Dibromomethane	93	95, 174
1,2-Dichlorobenzene	146	111, 148
1,3-Dichlorobenzene	146	111, 148
1,4-Dichlorobenzene	146	111, 148
trans-1,4-Dichloro-2-Butene	53	89, 75
Dichlorodifluoromethane	85	87
1,1-Dichloroethane	63	65, 83
1,2-Dichloroethane	62	98
1,1-Dichloroethene	96	61, 63
cis-1,2-Dichloroethene	96	61, 98
Trans-1,2-Dichloroethene	96	61, 98
1,2-Dichloropropane	63	112
1,3-Dichloropropane	76	78
2,2-Dichloropropane	77	97
1,1-Dichloropropene	75	110, 77
1,4-Dioxane	88	58
Ethyl Acetate	45	70
Ethylbenzene	91	106
Ethyl Ether	59	45, 74
Ethyl Methacrylate	69	41
Hexane	57	41,86
2-Hexanone	43	58,57
Hexachlorbutadiene	225	223, 227
Isobutyl Alcohol	43	74
Isopropylbenzene	105	120
p-Isopropyltoluene	119	134, 91
Methacrylonitrile	67	41
Methylene Chloride	84	86, 49
Methyl Iodide	142	127, 141
Methyl Methacrylate	69	41
4-Methyl-2-Pentanone	43	58
Naphthalene	128	—
2-Nitropropane	43	41
Pentachloroethane	167	169
Propionitrile	54	52
n-Propylbenzne	91	120
Styrene	104	78
1,1,1,2-Tetrachloroethane	131	133, 119
1,1,2,2-Tetrachloroethane	83	131, 85
Tetrachloroethene	166	168, 129
Toluene	92	91
Trans-1,3-Dichlorpropene	75	77
1,2,3-Trichlorobenzene	180	182, 145
1,2,4-Trichlorobenzene	180	182, 145
1,1,1-Trichloroethane	97	99, 61
1,1,2-Trichlorethane	83	97, 85
Trichloethene	95	130, 132
Trichlorofluoromethane	101	103
1,2,3-Trichloropropane	75	77
1,2,4-Trimethylbenzene	105	120
1,3,5-Trimethylbenzene	105	120
Vinyl Chloride	62	64
o-Xylene	106	91
m,p-Xylene	106	91
Vinyl Acetate	43	86

Table 3
BFB Key Ions and Abundance Criteria

Mass	Ion Abundance Criteria
50	15.0 to 40% of mass 95
75	30 to 60% of mass 95
95	Base Peak, 100% Relative Abundance
96	5 to 9% of mass 95
173	<2% of mass 174
174	>50% of mass 95
175	5 to 9% of mass 174
176	>95% but <101% of mass 174
177	5 to 9% of mass 176

Table 4
Surrogate Recovery Limits*

Surrogate Compound	Water	Low/Med Soil/Sediment
4-Bromofluorobenzene	86-115	74-121
Dibromofluoromethane	86-118	80-120
Toluene-d_8	88-110	81-117
1,2-Dichloroethane-d_4	80-120	80-120

*Based upon method limits

Table 5
Volatile Internal Standards with Corresponding Analytes Assigned for Quantitation

Pentafluorobenzene	Chlorobenzene-d5	1,4-Difluorobenzene	1,4-Dichlorobenzene-D4
Acetone	Bromoform	Benzene	Bromobenzene
Acetonitrile	Chlorobenzene	Bromodichloromethane	n-Butylbenzene
Acrolein	1-Chlorohexane	Bromofluorobenzene (Surrogate)	sec-Butylbenzene
Acrylonitrile	Cyclohexanone	Carbon Tetrachloride	tert-Butylbenzene
Allyl Chloride	trans-1,4-Dichloro-2-Butene	2-Chloroethylvinylether	2-Chlorotoluene
Bromochloromethane	1,3-Dichloropropane	1,2-Dibromoethane	4-Chlorotoluene
Bromomethane	Ethylbenzene	1,2-Dichloroethane	1,2-Dibromo-3-Chloropropane
2-Butanone	2-Hexanone	Dibromofluoromethane (Surrogate)	1,2-Dichlorobenzene
Carbon Disulfide	Pentachloroethane	1,2-Dichloropropane	1,3-Dichlorobenzene
Chloroethane	Styrene	1,1-Dichloropropene	1,4-Dichlorobenzene
Chloroform	1,1,1,2-Tetrachloroethane	cis-1,3-Dichloropropene	Hexachlorobutadiene
Chloromethane	Tetrachloroethene	trans-1,3-Dichloropropene	Isopropyl benzene
Chloroprene	Dibromochloromethane	1,4-Dioxane	p-Isoproyltoluene
Dichlorodifluoromethane	m,p-Xylene	Ethyl Methacrylate	Naphthalene
1,1-Dichloroethane	o-Xylene	Isobutyl Alcohol	n-Propylbenzene
1,1-dichloroethene		4-Methyl-2-Pentanone	1,1,2,2-Tetrachloroethane
cis-1,2-Dichloroethene		Methyl Methacrylate	1,2,3-Trichlorobenzene
trans-1,2-Dichloroethene		2-Nitropropane	1,2,4-Trichlorobenzene
2,2-Dichloropropane		Toluene	1,2,3-Trichloropropane
Ethyl Acetate		Toluene-d_8 (Surrogate)	1,2,4-Trimethylbenzene
Ethyl Ether		1,1,2-Trichloroethane	1,3,5-Trimethylbenzene
Iodomethane		Trichloroethene	
Mathacrylonitrile		1,2-Dichloroethane-d_4 (Surrogate)	
Methylene Chloride			
Methyl Iodide			
Propionitrile			
1,1,1-Trichloroethane			
Trichlorofluoromethane			
Vinyl acetate			
Vinyl chloride			

Typist: LLR

Table 6
Volatile Calibration Check Compounds (CCC)

- 1,1-Dichloroethene
- Toluene
- Chloroform
- Ethylbenzene
- 1,2-Dichloropropane
- Vinyl Chloride

Initial Calibration: 5,20,50,100,200ppb (soil/5mL purge)
Max%RSD: 30%
Max%Difference: 20%
Calibration Verification: 50ppb (soil/5mL purge)

Volatile System Performance Check Compounds (SPCC)

- Chloromethane
- 1,1-Dichloroethane
- Bromoform
- 1,1,2,2-Tetrachloroethane
- Chlorobenzene

Minimum Relative Response Factor (RRF)
Soil /5mL Purge RRF:

0.100 for Bromoform
1,1-Dichloroethane
Chloromethane
0.300 for Chlorobenzene
1,1,2,2-Tetrachloroethane

Table 7
Compound List and Method Reporting Limits

Volatile Compound	CAS#	5mLWater or 5g Soil*	Volatile Compound	CAS#	5mL Water or 5g Soil*
Acetone	67-64-1	5	1,2-Dichloropropane	78-87-5	5
Benzene	71-43-2	5	cis-1,3-Dichloropropene	10061-01-5	5
Bromobenzene	108-86-1	5	trans-1,3-Dichloropropene	10061-02-6	5
Bromochloromethane	74-97-5	5	1,3-Dichloropropane	142-28-9	5
Bromodichloromethane	75-27-4	5	2,2-Dichloropropane	594-20-7	5
Bromoform	75-25-2	5	1,1-Dichloropropene	63-58-6	5
Bromomethane	71-83-9	5	Ethylbenzene	100-41-4	5
2-Butanone	78-93-3	5	2-Hexanone	591-78-6	5
Carbon Disulfide	75-15-0	5	Hexachlorobutadiene	87-68-3	5
n-Butylbenzene	104-51-8	5	Isopropylbenzene	92-82-8	5
sec-Butylbenzene	135-98-8	5	p-Isopropyltoluene	99-87-6	5
tert-Butylbenzene	98-06-6	5	Methylene chloride	75-09-2	5
Carbon Tetrachloride	56-23-5	5	4-Methyl-2-Pentanone	108-10-1	5
Chlorobenzene	108-90-7	5	Methyl-tert-butyl Ether	1634-04-4	5
Chloroethane	75-00-3	5	Naphthalene	91-20-3	5
Chloroform	67-66-3	5	n-Propylbenzene	103-65-1	5
2-Chloroethylvinyl Ether	110-75-8	5	Styrene	100-42-5	5
Chloromethane	74-87-3	5	1,1,1,2-Tetrachloroethane	630-20-6	5
2-Chlorotoluene	95-49-8	5	1,1,2,2-Tetrachloroethane	79-34-5	5
4-Chlorotoluene	106-43-4	5	Tetrachloroethene	127-18-1	5
Dibromochloromethane	124-48-1	5	Toluene	108-88-3	5
1,2-Dibromo-3-Chloropropane	92-12-8	5	1,2,3-Trichlorobenzene	87-61-6	5
1,2-Dibromoethane	106-93-4	5	1,2,4-Trichlorobenzene	120-82-1	5
Dibromomethane	74-94-3	5	1,1,1-Trichloroethane	71-55-6	5
1,2-Dichlorobenzene	95-50-1	5	1,1,2-Trichloroethane	79-00-5	5
1,3-Dichlorobenzene	541-73-1	5	Trichloroethene	79-01-6	5
1,4-Dichlorobenzene	106-46-7	5	Trichlorofluoromethane	75-69-4	5
Dichlorodifluoromethane	75-71-8	5	1,1,2-Trichlorotriflouroethane	76-13-1	5
1,1-Dichloroethane	75-34-3	5	1,2,3-Trichloropropane	96-18-4	5
1,2-Dichloroethane	107-06-2	5	1,2,4-Trimethylbenzene	95-63-6	5
1,1-Dichloroethene	75-35-4	5	1,3,5-Trimethylbenzene	108-67-8	5
cis-1,2-Dichloroethene	156-59-2	5	Vinyl Chloride	75-01-4	5
trans-1,2-Dichloroethene	156-60-5	5	o-Xylene	95-47-6	5
1,2-Dichloroethene (Total)	540-59-0	5	m,p-Xylene	13-302-07	5
			Xylene (Total)	1330-20-7	5
			Vinyl Acetate	108-05-4	5

NOTE: Concentration Units: Water = µg/L Soil = µg/Kg
*Reporting Limits derived from method.

Table 8
Standards Preparation—Soil/5mL Purge

Compound or Standard Mix	Begin Conc. (µg/mL)	Aliquot (µL)	Total Vol. (mL)	Final Conc. (µg/mL)
Internal Standard				
NSI:				
Internal Standard Mix	2500	200	20	25
Surrogate:				
NSI:				
Surrogate Mix	2500	200	20	25
VSTD025				
NSI:				
Mix A	2000	125	10	25
Mix C	2000	125	10	25
Mix L	2000	125	10	25
Mix M	2000	125	10	25
Carbon Disulfide	5000	50	10	25
Vinyl Acetate	5000	50	10	25
Ketones Mix	1000	125	10	25
Extra Compound Mix				
Chem-Service:				
2-Chloroethylvinyl Ether	10,000	25	10	25
Acrolein	10,000	250	10	250
Acrylonitrile	10,000	250	10	250
1-Chlorohexane	10,000	25	10	25
MTBE	10,000	25	10	25
Methyl Iodide	10,000	25	10	25
Trans-1,4-Dichloro-2-Butene	10,000	25	10	25
n-Hexane	10,000	25	10	25
Acetonitrile	10,000	250	10	250
1,1,2-Trichlorotrifluoroethane	10,000	25	10	25
Allyl Chloride	10,000	25	10	25
Chloroprene	10,000	25	10	25
Propionitrile	10,000	250	10	250
Methacrylonitrile	10,000	250	10	250
Isobutyl Alcohol	10,000	500	10	500
1,4-Dioxane	10,000	500	10	500
Methyl Methacrylate	10,000	25	10	25
Ethyl Methacrylate	10,000	25	10	25
Pentachloroethane	10,000	25	10	25
2-Nitropropane	10,000	25	10	25
Cyclohexanone	10,000	25	10	25
Ethyl Acetate	10,000	25	10	25
n-Butanol	10,000	250	10	250
Ethyl Ether	10,000	25	10	25
Gases Mix				
NSI:				
Gases Mix	1000	125	5	25

Typist: LLR

Table 9

Response Factor and Concentration Calculation

$$RF = \frac{(A_x)\ (C_{is})}{(A_{is})\ (C_x)} \quad \text{and} \quad conc_{RF} = \frac{(A_x)\ (C_{is})}{(A_{is})\ (\overline{RF})} \quad \text{or} \quad conc_{LR} = \frac{(A_x) - b}{M} \quad \text{or} \quad conc_{quad} = a + bx + cx^2$$

Where:

A_x = Area of characteristic ion for the measured compound.
A_{is} = Area of the characteristic ion for the specified internal standard
C_{is} = Concentration of the internal standard (ng/µL)
C_{RF} = Concentration of the measured compound (ng/µL) using the average response factor
$\overline{RF}$ = Average of Five Initial RF's for a compound
C_{LR} = Concentration of the measured compound (ng/µL) using Linear Regression
C_{quad} = Concentration of the measured compound (ng/µL) using the Quadratic Equation

---

Surrogate Recovery: $\% \text{ Surrogate Recovery} = \frac{Qd}{Qa} \times 100\%$

Where:

Qd = Quantity determined by analysis
Qa = Quantity added to sample

Matrix Spike recovery and RPD:

$$\text{Matrix Spike Recovery} = \frac{SSR - SR}{SA} \times 100 \qquad \text{Matrix Spike RPD} = \frac{|MSR - MSDR|}{(½)(MSR + MSDR)} \times 100$$

Where:

SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

Laboratory Control Spike (LCS) Recovery: $\text{LCS Spike Recovery} = \frac{SSR - SR}{SA} \times 100$

Where:

SSR = Spiked Sample Result
SR = Sample Result
SA = Spike Addeds

Calibration Calculations:

$$\%RSD = \frac{SD}{\overline{RF}} \times 100\% \qquad \%Drift = \frac{RF_v - \overline{RF}}{\overline{RF}} \times 100$$

Where:

SD = Standard Deviation
$\overline{RF}$ = Average of Five Initial RF's for a compound
RSD = Relative Standard Deviation

Where:

RF_v = RF_v from Continuing Calibration
$\overline{RF}$ = The mean Response Factor from the Initial Calibration

Table 10
Additional 8260 Standards Final Concentration—Soil/5mL Purge

Compound in Standard Mix	Final Conc. 5ppb Std	Final Conc. 20ppb Std	Final Conc. 50ppb Std	Final Conc. 100ppb Std	Final Conc. 200ppb Std
Acetonitrile	50	200	500	1000	2000
Acrolein	50	200	500	1000	2000
Acrylonitrile	50	200	500	1000	2000
Allyl Chloride	5	20	50	100	200
n-Butanol	100	400	1000	2000	4000
1-Chlorohexane	5	20	50	100	200
Chloroprene	5	20	50	100	200
Cyclohexanone	5	20	50	100	200
Trans-1,4-Dichloro-2-Butene	5	20	50	200	400
1,4-Dioxane	100	400	1000	2000	4000
Ethyl Acetate	5	20	50	100	200
Ethyl Ether	5	20	50	100	200
Ethyl Methacrylate	5	20	50	100	200
Hexane	5	20	50	100	200
Isobutyl Alcohol	100	400	1000	2000	4000
Methacrylonitrile	50	200	500	1000	2000
Methyl Iodide	5	20	50	100	200
Methyl Methacrylate	5	20	50	100	200
2-Nitropropane	5	20	50	100	200
Pentachloroethane	5	20	50	100	200
Propionitrile	50	200	500	1000	2000

Table 11
Skinner's Compound List and Method Reporting Limits (MS330)

Volatile Compound	CAS#	5mLWater or 5g Soil*
Benzene	71-43-2	5
2-Butanone	78-93-3	5
Carbon Disulfide	75-15-0	5
Chlorobenzene	108-90-7	5
Chloroform	67-66-3	5
1,2-Dibromoethane	106-93-4	5
1,2-Dichloroethane	107-06-2	5
1,4-Dioxane	123-91-1	500
Ethylbenzene	100-41-4	5
Sytrene	100-42-5	5
Xylene (Total)	1330-20-7	5

NOTE: Concentration Units: Water=µg/L Soil=µg/Kg

*Reporting Limits derived from method. Medium Soil/Sediment Method Reporting Limits are 125 times the individual Low Soil/Sediment Method Reporting Limits.

Table 12
TCLP** Compound List and Method Reporting Limits (MS340)

Volatile Compound	CAS#	5mLWater µg/L	Regulatory Limits (mg/L)
Benzene	71-43-2	5	0.50
2-Butanone	78-93-3	5	200
Carbon Tetrachloride	56-23-5	5	0.50
Chlorobenzene	108-90-7	5	100
Chloroform	67-66-3	5	6
1,2-Dichloroethane	107-06-2	5	0.50
1,1-Dichloroethene	75-35-4	5	0.70
Tetrachloroethene	79-34-5	5	0.70
Trichloroethene	79-00-5	5	0.50
Vinyl Chloride	108-67-8	5	0.20

**TCLP extracts must be analyzed within 14 days of the zero headspace extraction date.
The TCLP Zero Headspace Extraction Blank is analyzed with no dilution, resulting in the above reporting limits. All samples are analyzed initially at a 5X dilution to reduce the effects of the acetic acid on the column. The higher reporting limits as a result of the dilution are still below the regulatory limits.

Table 13
Appendix IX Compound List and Method Reporting Limits (MS350)

Volatile Compound	CAS#	5mLWater or 5g Soil*	Volatile Compound	CAS#	5mL Water or 5g Soil*
Acetone	67-64-1	5	Ethyl Methacrylate	97-63-2	5
Acetonitrile	75-05-8	200	Ethylbenzene	100-41-4	5
Acrolein	107-02-8	50	2-Hexanone	591-78-6	5
Acrylonitrile	107-13-1	50	Isobutyl Alcohol	78-83-1	200
Allyl Chloride	107-05-1	5	Methacrylonitrile	126-98-7	100
Benzene	71-43-2	5	Methyl Iodide	74-88-4	5
Bromobenzene	108-86-1	5	Methyl Methacrylate	80-62-6	5
Bromodichloromethane	75-27-4	5	Methylene chloride	75-09-2	5
Bromoform	75-25-2	5	4-Methyl-2-Pentanone	108-10-1	5
Bromomethane	71-83-9	5	Pentachloroethane	76-01-75	5
2-Butanone	78-93-3	5	Propionitrile	107-12-0	100
Carbon Disulfide	75-15-0	5	Styrene	100-42-5	5
Carbon Tetrachloride	56-23-5	5	1,1,1,2-Tetrachloroethane	630-20-6	5
Chlorobenzene	108-90-7	5	1,1,2,2-Tetrachloroethane	79-34-5	5
Chloroethane	75-00-3	5	Tetrachloroethene	127-18-1	5
2-Chloroethylvinyl Ether	110-75-8	5	Toluene	108-88-3	5
Chloroform	67-66-3	5	Trans-1,4Dichloro-2-Butene	110-57-6	5
Chloromethane	74-87-3	5	Trans-1,3-Dichloropropene	10061-02-6	5
Chloroprene	126-99-8	5	1,2,4-Trichlorobenzene	120-82-1	5
Cis-1,3-Dichloropropene	10061-01-5	5	1,1,1-Trichloroethane	71-55-6	5
1,2-Dibromo-3-Chloropropane	96-12-8	5	1,1,2-Trichloroethane	79-00-5	5
Dibromochloromethane	124-48-1	5	Trichloroethene	79-01-6	5
1,2-Dibromoethane	106-93-4	5	Trichlorofluoromethane	75-69-4	5
Dibromomethane	74-94-3	5	1,2,3-Trichloropropane	96-18-4	5
Dichlorodifluoromethane	75-71-8	5	Vinyl Chloride	75-01-4	5
1,1-Dichloroethane	75-34-3	5	Xylene (Total)	1330-20-7	5
1,2-Dichloroethane	107-06-2	5	Vinyl Acetate	108-05-4	5
1,1-Dichloroethene	75-35-4	5			
1,2-Dichloroethene (Total)	540-59-0	5			
1,2-Dichloropropane	78-87-5	5			
1,4-Dioxane	123-91-1	500			
trans-1,3-Dichloropropene	10061-02-6	5			

NOTE: Concentration Units: Water=µg/L Soil=µg/Kg

*Reporting Limits derived from method. Medium Soil/Sediment Method Reporting Limits are 125 times the individual Low Soil/Sediment Method Reporting Limits.

Typist: LLR

Table 14
BTEX+ Compound List and Method Reporting Limits (MS380)

Volatile Compound	CAS#	5mLWater or 5g Soil*
Benzene	71-43-2	5
Ethylbenzene	100-41-4	5
Toluene	108-88-3	5
Xylene (Total)	1330-20-7	5
MTBE	1634-04-4	5
Hexane	110-54-3	5
1,2-Dibromoethane	106-93-4	5

NOTE: Concentration Units: Water=µg/L Soil=µg/Kg

*Reporting Limits derived from method. Medium Soil/Sediment Method Reporting Limits are 125 times the individual Low Soil/Sediment Method Reporting Limits.

ATTACHMENT 1
Volatiles GC/MS pH Log

VOLATILES
pH FORM

Ref: SWL-OV-101, Rev. 2.0
Form ID: OV-101-pH-F
Location/File ID: QCFORMS ON 'SWLO'\ORGANICS\PHLOG.DOC

CLIENT _____ SDG/EPISODE# _____

LAB ID	CLIENT SAMPLE ID	pH	ANALYST	DATE TESTED	DATE P.O. NOTIFIED*

COMMENTS: _____

* Date P.O. Notified: Field is filled out when samples are not acidified and Project Officer is notified of situation

SOUTHWEST LABORATORY OF OKLAHOMA, INC./AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.
1700 W. ALBANY, BROKEN ARROW, OK 74012 (918)251-2858

Typist: LLR

ATTACHMENT 2
Encore Sample Preservation Form

VOLATILES Method 5035
Encore Sample Preservation Form

Ref: SWL-OV-303, Rev. 4.1
Form ID: OV-303-Prsv-1
Location/File ID: S:\COMMON\ORGANICS\5035.DOC

CLIENT _____ EPISODE# _____

VOA BALANCE ID A B C (Circle One)

MeOH LOT #_____ $NaHSO_4$ LOT _____

LAB ID/ENCORE ID	WEIGHT (g)	ANALYST	DATE PREPARED

SOUTHWEST LABORATORY OF OKLAHOMA, INC./AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.
1700 W. ALBANY, BROKEN ARROW, OK 74012 (918)251-2858

ATTACHMENT 3
VOA Vial Preservation/Preparation Form

VOLATILES Method 5035
VOA Vial Preservation/Preparation Form

Ref: SWL-OV-303, Rev. 4.1
Form ID: OV-303-Prep-2
Location/File ID: S:\COMMON\ORGANICS\5035prep.DOC

CLIENT:_____ EPISODE #:_____

VOA BALANCE ID: A B C (Circle One)

MeOH LOT #:_____ $NaHSO_4$ LOT:_____

DATE: _____

LAB ID/ VOA VIAL ID*	INITIAL WEIGHT (g)**	FINAL WEIGHT (g)	ANALYST

*Assigned upon receipt at the laboratory after sampling.
**Performed before vials are submitted to client. Weight noted directly on VOA vial label.

SOUTHWEST LABORATORY OF OKLAHOMA, INC./AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.
1700 W. ALBANY, BROKEN ARROW, OK 74012 (918)251-2858

Typist: LLR

PPENDIX A—SW-846 METHOD 5035 FOR VOLATILES

1.0 SCOPE AND APPLICATION

1.1 The analytical method that follows is designed to analyze soil samples from sites for volatile compounds based on a closed-system purge and trap GC/MS method.

1.2 The reporting limits and compounds listed in Table 7 are normal limits and compounds analyzed by this method.

2.0 METHOD SUMMARY

2.1 Low-level volatile organic compounds are determined by collecting approximately 5g of sample, weighed in the field at the time of collection, and placing it in a pre-weighed vial with a septum-sealed screw-cap that already contains a stirring bar and 5mL of sodium bisulfate preservative solution. The entire vial is placed, unopened, onto the instrument. Immediately before analysis, organic-free reagent water, surrogates, and internal standards are automatically added without opening the sample vial.

2.2 The sample introduction technique in Appendix Section 2.1 is not applicable to medium-level samples. Medium-level volatile organic compounds are determined by collecting approximately 5g of sample, weighed in the field at the time of collection, and placing it in a pre-weighed vial with a septum-sealed screw cap that already contains a stirring bar and 10mL of methanol preservative. Analysis of the medium level sample proceeds as stated earlier in the SOP in Section 8.

2.3 An alternative procedure to submitting sample vials to the laboratory is the submission of Encore Samplers. Transfer of the sample into the appropriate preservative is then performed at the laboratory.

3.0 HEALTH AND SAFETY

3.1 Some of the reagents solvents, and other chemicals used in this method have been clearly defined as toxic and/or carcinogenic. Most, however, remain in question or are not precisely defined with respect to the hazards of handling. All chemicals and samples, therefore, are treated as potentially hazardous, and exposure to them is minimized. Also, the MSDS for all stock solutions, reagents, and solvents used are kept on file in an easily accessible place in the lab. Any spills that may occur are cleaned up immediately.

4.0 SAMPLE PRESERVAION, CONTAINERS, HANDLING & STORAGE

4.1 All soil samples are to be preserved, whether it is performed in the field or in the laboratory.

4.2 Samples are stored at 4 +/- 2 C. All samples must be allowed to warm to ambient temperature before analysis.

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

5.1 The same sources of contamination stated in Section 5.0 of this SOP apply to Method 5035.

6.0 APPARATUS AND MATERIALS

6.1 Purge and Trap Device---The purge and trap device consists of the sample purger, the trap, and the desorber. The sample purger must be capable of analyzing a soil by means of a closed-system autosampler. It must also have the capability of automatically adding internal standard and surrogate without opening the sample vial. Sample purgers include the Tekmar Precept II and the Varian Archon.

6.2 Magnetic Stirring Bars—PTFE- or glass coated bars of the appropriate size to fit in the sample vials. Stirring bars may be reused, provided they are thoroughly cleaned between uses.

6.3 Encore Sample Extractor---Metal device used to extract soil sample out of Encore Samplers into VOA vials.

7.0 REAGENTS AND STANDARDS

7.1 Reagent water — Produced by filtering tap water through an organic filtration system containing activated charcoal prior to use.

7.2 Methanol — Pesticide quality or equivalent.

7.3 Secondary dilution standards — Using commercially prepared standard solutions, secondary dilution standards that contain the compounds of interest, either singly or mixed together, are prepared in methanol. Secondary dilution standards should be stored with minimal headspace and should be checked frequently for signs of degradation or evaporation especially just prior to preparing calibration standards from them. Secondary dilution standards are used until degradation is verified through daily calibration checks.

7.4 Internal Standard and Surrogate Spiking Solution — The internal standards are Pentafluorobenzene, 1,4-Difluorobenzene, Chlorobenzene-d_5, and 1,4-Dichlorobenzene-d_4. The surrogates are Dibromofluoromethane, Toluene-d_8, 4-Bromofluorobenzene, and 1,2-Dichloroethane d_4. One internal standard and surrogate standard solution is prepared containing the above at a concentration of 250 µg/mL in methanol.

7.5 Sodium Bisulfate—ACS reagent grade or equivalent

8.0 PROCEDURE

8.1 Sample Containers: Clients may submit samples to the laboratory in one of two ways; In preserved 40mL VOA vials or in a device such as the Encore sample.

8.1.1 Preserved 40mL VOA Vials

Typist: LLR

8.1.1.1 Before sampling, the 40mL VOA vials must be prepared. This preparation may be performed by our laboratory or the client responsible for sampling. The specific preparation procedure depends on the expected concentration range of the sample. If the concentration range is unknown, enough sample should be taken to prepare both low and high concentration vials.

8.1.1.2 Sample vials should be prepared in a controlled environment, sealed, and shipped to the field location. The vials containing preservative for low concentration soil samples should be prepared by the following steps:

- Add a clean magnetic stirring bar to each clean vial.
- Add 1g of NaHSO4 preservative to each vial.
- Add 5mL of organic-free reagent water to each vial.
- Seal the vial with the screw-cap and septum seal.
- Affix a label to each vial.
- Weigh the prepared vial to the nearest 0.01g, record the weight, and write it on the label.

8.1.1.3 The vials containing preservative for high concentration soil samples should be prepared using the following steps:

- Add 10mL of methanol to each vial.
- Seal the vial with the screw-cap and septum seal.
- Affix a label to each vial.
- Weigh the prepared vial to the nearest 0.01g, record the weight, and write it on the label.

8.1.1.4 Pre-preserved vials may also be used. These vials are obtained from a commercial vendor.

8.2 Sample Collection: Samples may be collected in either an Encore sampler or in preserved VOA vials.

8.3 Sample Storage:

8.3.1 If samples arrive at the laboratory in Encore Samplers, they must be analyzed or preserved within 48 hours of sampling. Preservation is performed in the same manner as stated above, depending on the expected sample concentration level.

8.3.2 The sample core is removed from the Encore Sampler and added to the vial (Attachment 3.)

8.3.3 - The vial is weighed again to determine the precise weight of the sample, and the Encore Preservation Form is prepared. This form is submitted with sample data.

8.3.4 If the samples arrive at the laboratory in pre-preserved vials, the VOA Vial Preservation/Preparation Form (Attachment 1) is prepared. The initial weight of the vial and preservation (which was determined by the client or commercial vendor) is noted in the Initial Weight column. The vial containing the sample is weighed and this weight is noted in the Final Weight column. The difference between the Final Weight and Initial Weight is the weight of the sample, which is used for calculations.

8.4 Instrument Requirements: Low level samples requiring Method 5035 must be analyzed on the Tekmar Precept or Varian Archon to accommodate the "closed-system" requirements.

8.5 All other calibration and sample analysis procedures would be performed as stated in Sections 8.0 through Sections 12.0 of the SOP, with specific regards to the Tekmar Precept, Varian Archon, or equivalent.

Typist: LLR

SOUTHWEST LABORATORY OF OKLAHOMA, INC. *and*
AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.

TM

Standard Operating Procedure

Analysis of Volatile Organics by GC/MS 8260B — 25mL Purge

Document No.: SWL-OV-304

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APPROVALS	DATES
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GC/MS ANALYSIS OF PURGEABLE ORGANICS

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GC/MS ANALYSIS OF PURGEABLE ORGANICS (25mL PURGE)

1.0. SCOPE AND APPLICATION

1.1 Method 8260A, test code MS328, is a purge-and-trap, gas chromatograph/mass spectrometer procedure used to determine volatile organic compounds in low level water samples. It can be used to quantitate most volatile organic contaminants with boiling points less than 200°C that are insoluble or only slightly soluble in water. It can also be used for the quantitation and analysis of the more soluble organic compounds such as ethers, acetates, acrylates, and ketones, but with quantitation limits increased accordingly due to poor purging efficiency. In general, for most contaminants, the reporting limit for this method for any one compound is 1 µg/L. Clearly, reporting limits will be higher for samples requiring dilution due to high concentrations of contaminants.

1.2 The reporting limits and compounds listed in Table 7 are normal limits and compounds analyzed by this method.

2.0 METHOD SUMMARY

2.1 An inert gas, helium, is bubbled through either an aqueous contained in a specifically designed purging chamber at ambient temperature. The purgeables are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the purgeables are trapped. After purging is completed, the sorbent column is heated and backflushed with the helium to desorb the purgeables onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the purgeables which are then detected with a mass spectrometer.

2.2 For normal samples, a 25mL aliquot is used.

2.3 For dilutions, an aliquot of the sample is diluted with filtered tap water. A 25 mL aliquot of the dilution is taken for purging. If the amount of a compound detected is outside the linear range of the instrument, then a smaller sample size is analyzed.

2.4 All samples are analyzed within 14 days of the sampling date. (See Section 4.1 for details concerning sample preservation)

3.0 HEALTH & SAFETY

Some of the reagents solvents, and other chemicals used in this method have been clearly defined as toxic and/or carcinogenic; most, however, remain in question or are not precisely defined with respect to the hazards of handling. All chemicals and samples, therefore, are treated as potentially hazardous, and exposure to them is minimized. Also, the MSDS for all stock solutions, reagents, and solvents used are kept on file in an easily accessible place in the lab. Any spills that may occur are cleaned up immediately.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING & STORAGE

4.1 All water samples are to be preserved in the field using HCl. Verification of this preservation is performed by checking the pH of all water samples and recording their pH on the Volatile GC/MS pH Form. (Figure 1)

4.2 Samples are stored at 4° +/- 2° C. Standards are stored at –10° to –20°C. All samples and standard solutions must be allowed to warm to ambient temperature before analysis.

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

5.1 Many major sources of contamination are avoided by using stainless steel, nickel, polytetrafluroethylene (PFTE), glass, and other inert materials for all equipment and materials that in any way affect the detection and quantitation of volatile contaminants in samples. However, other areas of contamination do exist, and are avoided as follows:

5.1.1 Samples can sometimes be contaminated by diffusion of volatile organics through the septum seal during shipment and storage; this can be handled by the use of a trip blank prepared from organic-free filtered tap water which is carried through the entire sampling and shipping process with all samples, in addition to holding blanks prepared in the same fashion and stored in all areas where samples will be stored in the laboratory. Our laboratory encourages the analysis of a trip blank and sends trip blanks to clients when providing a bottle order for sampling.

5.1.2 Interfering contamination can occur when a sample is analyzed immediately following a sample containing high concentrations of volatile organic compounds. All such samples are reviewed carefully for any indication of carryover and reanalyzed when there is any question of whether contaminants found in the sample are the result of true contamination or originate from the previous analysis.

5.1.3 Common laboratory solvents present in the air of most analytical laboratories, notably methylene chloride, acetone, and chloroform, can cause contamination of the analytical instruments themselves; this can be checked by analyzing an instrument blank, where the presence of volatile contaminants can alert the analyst of the need to perform any cleaning measures necessary to achieve an analytical run which is as contaminant-free as possible.

6.0. APPARATUS AND MATERIALS

6.1 Micro syringes — 10 µL and larger, 0.006 inch ID needle.

6.2 Syringe valve — two-way, with Luer ends (three each), if applicable to the purging device.

6.3 Syringe — 25 mL, gas tight.

6.4 pH Paper — Range of 1-6

6.5 Balance, Analytical — capable of accurately weighing 0.0001 g. and a top-loading balance capable of weighing 0.1 g.

6.6 Glassware

6.6.1 Bottle — 40 mL, screw cap, with Teflon cap liner.

6.6.2 Volumetric flasks — class A with ground-glass stoppers.

6.6.3 Vials — 20mL for medium level extraction

6.7 Purge and trap device — The purge and trap device consists of three separate pieces of equipment; the sample purger, trap and the desorber. Heating blankets are added to the Tekmar 2032 and 2016 Autosamplers when low level soils are analyzed. Sample purgers include Tekmar ALS2032, ALS2016, Precept or Archon, or equivalent. Trap and desorber include Tekmar 2000 and 3000 or equivalent.

6.8 Gas chromatograph — An analytical system complete with a temperature programmable gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical column, and gases. GC is Hewlett-Packard 5890 Series II or equivalent.

6.9 Column — DB624, 75m, 0.53mm ID, 3.0 um (J&W Scientific Cat. No. 125-1334).

6.10 Jet Separator — Glass (SIS Cat. No. 113505).

6.11 Mass spectrometer — Capable of scanning from 35 to 300 amu every one second, utilizing seventy volts (nominal) electron energy in the electron impact ionization mode and producing a mass spectrum which meets all the criteria when 50 ng of 4-Bromofluorobenzene (BFB) is injected through the gas chromatograph inlet (see Table 3). MSD is Hewlett-Packard 5970, 5971, 5972, or equivalent.

6.12 Calibration requirements can be found in Table 6.

7.0 REAGENTS AND STANDARDS

7.1 Reagent water — Produced by filtering tap water through an organic filtration system containing activated charcoal prior to use.

7.2 Methanol — Pesticide quality or equivalent.

7.3 Stock standard solutions — Stock standard solutions are purchased and are traceable to EPA supplied standards. These solutions are stored in Teflon sealed screwcap bottles at -10°C to -20°C.

7.4 Secondary dilution standards — Using commercially prepared standard solutions, secondary dilution standards that contain the compounds of interest, either singly or mixed together, are prepared in methanol. Secondary dilution standards should be stored with minimal headspace and should be checked frequently for signs of degradation or evaporation especially just prior to preparing calibration standards from them. Secondary dilution standards are used until degradation is verified through daily calibration checks.

7.5 Internal Standard and Surrogate Spiking Solution — The internal standards are Pentafluorobenzene, 1,4-Difluorobenzene, Chlorobenzene-d_5, and 1,4-Dichlorobenzene-d_4. The surrogates are Dibromofluoromethane, Toluene-d_8, 4-Bromofluorobenzene, and 1,2-Dichloroethane- d_4. Separate internal standard and surrogate standard solutions are prepared containing the above at a concentration of 25 µg/mL in methanol.

8.0 PROCEDURE

8.1 This method requires the following instrumental parameters:

8.1.1 Electron Energy: 70 Volts (nominal)

8.1.2 Mass Range: 35-300

8.1.3 Scan Time: to give at least 5 scans per peak, not to exceed one second per scan

8.2 Recommended Operating conditions for the gas chromatograph and column conditions: DB-624, 75m, 0.53mm ID, 3 µm film thickness at a flow rate of 30 mL/min. Column temperature is isothermal at 45°C for five minutes, then programmed at 5°C per minute to 105°C. further programmed at 10°C/min. to 180°C.

8.3 Adjust the purge gas (helium) flow rate to 35 ± 3 mL/min on the purge and trap. Variations from this flow rate may be necessary to achieve better purging and collection efficiencies for some compounds, particularly chloromethane and bromoform.

8.4 Tune

8.4.1 Before analysis of samples can begin, the instrument must meet all calibration criteria. The first step in the calibration of a Volatiles GC/MS system is to demonstrate the ionization and fragmentation of standard mass spectral tuning compounds. This, as well as a sensitivity check, is accomplished with the use of 4-Bromofluorobenzene, an EPA specified compound. This standard is run daily to validate the GC/MS system and to begin the 12-hour clock. BFB is analyzed by direct injection of a 50-ng aliquot into the instrument.

8.4.2 When the analysis is complete, the tune is checked against standard criteria (See Table 3 for Tune Criteria). SW8260 tune criteria for BFB must be met before any other calibration standards or sample analysis is performed.

8.4.3 If the tune fails to meet the criteria, BFB is rerun. If it fails again, the instrument must be retuned through "Auto Tune" or "Manual Tune," then BFB should be run again. If all criteria are met, the calibration sequence is run, but if it fails again, a supervisor is contacted because instrument maintenance such as source cleaning may be required.

8.5. Initial Calibration

8.5.1 Once BFB has met its tune criteria, calibration may begin. Initial calibration of the GC/MS is established and validated by the analysis of EPA traceable

standards at five concentration levels which are 1, 4, 10, 20, and 40 ppb. These standards are made up in 25 mL of filtered tap water and purged on the Tekmar before analysis by GC/MS. The internal standard calibration procedure is used. In addition to surrogate recovery compounds, standards and sample extracts are spiked with internal standards that span the retention time range of the analytes of interest.

8.5.2 The Response Factors (RF) for the analytes in the standard are calculated and stored in a Target Method File. Using the RFs from the initial calibration, the percent relative standard deviation (%RSD) for all compounds is calculated utilizing the formula shown in Table 9. The criterion the initial calibration must meet to be acceptable is found in Table 6.

8.5.3 If the five-point does not meet criteria, each standard is checked for the need of a possible rerun, in case the standards have been added at the wrong amount, or the purge may have been bad. If reanalyzing a standard will not solve the problem, a supervisor is contacted. Some problems could be found to be related to purge chambers, multiplier, flows, etc.

8.5.4 If the initial calibration does meet the criteria stated in Table 6, and there is time remaining in the 12-hour time period, samples may be run.

8.5.4.1 If the % RSD of any compound is 15% or less, then the relative response factor is assumed to be constant over the calibration range, and the average relative response factor may be used for quantitation.

8.5.4.2 If the % RSD of any compound is greater than 15%, a calibration curve must be constructed of area ratios (A/Ais) versus concentration using first order regression fit of the five calibration points (ie: linear regression).

The use of calibration curves is a recommended alternative to average response factor calibration and a useful diagnostic of standard preparation accuracy and absorption activity in the chromatographic system.

8.5.5 The calibration standards are prepared by filling a syringe with filtered tap water and spiking with the standards as follows.

25mL Purge --Tekmar ALS2032/ALS2016

Internal Standard (µL)	Surrogate (µL)	25ppm Stock Std. (µL)	8260 Extra Cmpd.Stock Standard (µL)	Gases Stock Std. (µL)	Int. Std. Final Conc. (ng/mL)	Standard Final Conc. (ng/mL)	Acrolein/ Acrylonitrile Final Conc. (ng/mL)
10	10	1	1	1	10	1	10
10	10	4	4	4	10	4	40
10	10	10	10	10	10	10	100
10	10	20	20	20	10	20	200
10	10	40	40	40	10	40	400

25mL Purge--Tekmar Precept or Varian Archon

25ppm Stock Std. (µL)	Gases Stock Std. (µL)	8260 Extra Cmpd. Stocks Standard (µL)	Int. Std. Final Conc. (ng/mL)	Standard Final Conc. (ng/mL)	Acrolein/ Acrylonitrile Final Conc. (ng/mL)
1.6	1.6	1.6	10	1	10
6.4	6.4	6.4	10	4	40
16	16	16	10	10	100
32	32	32	10	20	200
64	64	64	10	40	400

8.5.6 If analyzing on the Tekmar 2032/2016, the syringe is attached to the syringe valve assembly on the Tekmar. The syringe valve is opened, the sample is injected into the purging chamber, and the purging device valve is closed. Water samples are purged at ambient temperature. If analyzing on the Precept or Archon, the standard's 40mL volatile vial is loaded into the tray. The Precept or Archon will pick up the vial, withdraw 25mL from the vial, spike it with internal standard and surrogate, and transfer the 25ml to the Tekmar 3000 for purging. The sample is purged for 8.0 min at ambient temperature. At the conclusion of the purge time, the trapped materials are introduced to the GC column by the Desorb Mode where the trap is rapidly heated to 180°C for four minutes.

8.5.7 After the sample desorbs onto the GC, the trap is baked for four minutes at 225°C, then it is allowed to cool to less than 30°C before being ready to purge the next standard or sample. During this time, the Precept or Archon flushes its lines and needle.

8.5.8 The standard will be quantitated into a report in which concentrations, areas, response factors, standard deviations, percent differences, and regression coefficients are calculated.

8.6 Calibration Verification

8.6.1 A calibration verification of the GC/MS uses the same EPA traceable standards, but at only one level: 10 ppb. The analysis is performed in the same manner as stated before in the initial calibration. The calibration verification checks the same compounds as the initial calibration, and the criteria the calibration verification must meet to be acceptable are also found in Table 6.

All other compounds should have a %Drift less than 30%, except for the following which should have %Drift less than 60%: Freons, Ketones, 1,2,3-Trichloropropane, 1,2-Dibromo-3-Chloropropane, Naphthalene, Hexachlorobutadiene, and 2-Chloroethylvinylether.

8.6.2 The calibration verification is performed after the BFB and must meet all criteria before any other analysis continues. If the CCC/SPCC criteria are not met, the standard must be rerun, as it is possible, the standard was not prepared correctly or that there was a bad purge. However, if the second standard still doesn't meet CCC/SPCC criteria, an initial calibration must be run or corrective action performed.

8.6.3 If the calibration verification does meet the criteria stated in Table 6, samples may be run. Samples are quantitated based on the criteria stated above in Section 8.5.4.

8.7 Water Samples

8.7.1 Any headspace in the bottle is noted before opening. If analyzing on the Tekmar 2032 or 2016, the sample bottle (which has been allowed to come to ambient temperature) is opened and is carefully poured into the syringe barrel to just short of overflowing, then adjusted to a volume of 25 mL. This process of taking an aliquot destroys the validity of the sample for future analysis. Care must also be taken to prevent air from leaking into the syringe. The entire sample vial is loaded onto the autosampler.

8.7.2 When spiking samples on the Tekmar 2032 or 2016, 10 µL of the Surrogate Spiking Solution and 10 µL of the Internal Standard Spiking Solution are added through the valve bore of the syringe, which is equivalent to a concentration of 10 µg/L of each standard. If samples are analyzed on the Tekmar Precept or Varian Archon, the instrument automatically spikes Internal Standard and Surrogate during analysis.

8.7.3 Water samples are loaded in the following manner to ensure the proper sample is placed into its designated purge chamber on the Tekmar 2032/2016 Autosampler:

- A test tube rack is filled with the samples' 40 mL VOA vials. The syringes to be used are placed in front of the samples.
- All sample purge tubes are labeled with the SWLO ID Number and Autosampler position. Each analyst verifies as he/she loads each sample into the purge chambers the syringe matches the ID on the purge tube.
- The Precept or Archon allows the analyst to load the sample vials directly on the instrument, reducing the risk of a loading error. Purge in the same manner as stated during calibration.

8.7.4 For water samples analyzed on the Tekmar 2032 or 2016 requiring matrix spike and matrix spike duplicates, 10 µL of the matrix spike solution is added to the sample to be purged. Disregarding any dilutions, this is equivalent to a concentration of 10 µg/L of each matrix spike standard. Water samples analyzed on the Precept or Archon requiring MS/MSD, 16µL of the matrix spike solution is added to the sample to be purged, yielding a concentration of 10ug/L.

8.7.5 All dilutions should keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve.

8.7.6 An estimated concentration for Tentatively Identified Compounds (TICs) shall be quantified by the internal standard method. For quantification, the nearest internal standard shall be used.

8.7.7 The formula for calculating concentrations for target compounds is found in Table 9. For TICs, area counts from the total ion chromatograms are to be used for both the compound to be measured and the internal standard. A response factor (RF) of one (1) is to be assumed. The value from this quantitation shall be qualified as "estimated." This estimated concentration should be calculated for all TICs as well as those identified as unknowns.

8.7.8 After analysis is complete, the pH is recorded for all water samples on a pH Log (See Figure 1). If a sample has a pH reading >2, the project officer associated with that particular client's samples is notified.

8.8 Analysis Report Criteria--A sample is acceptable when it meets the following criteria:

8.8.1 The internal standards' EICP areas are within a factor of 2(-50%/+100%) of the calibration' internal standards.

8.8.2 The internal standards' retention times are within ±30 seconds of the calibration's internal standards.

8.8.3 Surrogate recoveries are within the limits stated in Table 4.

8.8.4 Any compound found in the sample is within the linearity range, with any dilutions showing compounds in an acceptable range of linearity. If a sample

analysis contains any target compounds above the linear range, all subsequent samples will be reviewed for those target compounds. The target compounds must be below the reporting limits to be acceptable; otherwise they must be reanalyzed.

8.8.5 Chromatogram shows no signs of carryover or instrument deficiencies.

8.9 Reanalysis Criteria: If sample does not meet internal standards' or surrogates' criteria, it must be reanalyzed. If the reanalysis meets all criteria, it is submitted. If the reanalysis duplicates the results from the first analysis, both analyses are submitted. This information is recorded in the analyst's logbook (See Figure 2).

8.10 A sample report is complete when it contains all of the following in order:

8.10.1 Chromatogram

8.10.2 Quant Report

8.10.3 Manual Integrations

8.10.4 Spectra of any compound, excluding internal standards and surrogates

8.10.5 Tentatively Identified Compounds (TICs)

8.10.6 Internal Standards/Surrogates

9.0 CALCULATIONS

9.1 All calculations are shown in Table 9 on page 19 of this SOP.

10.0. QUALITY ASSURANCE/QUALITY CONTROL

10.1 Method Blank (Laboratory Reagent Blank)

10.1.1 The method blank is analyzed once the calibration has been met, whether it be an initial calibration or calibration verification. The method blank, called the "VBLK", consists of loading a syringe with 25 mL of filtered tap water spiked to yield a concentration of 10 ppb internal standard and surrogate, or loading a 40mL volatile vial of filtered tap water into the Precept or Archon tray to be spiked automatically by the instrument. The VBLK is used to establish the analytical system is free of contaminants. It is also used to check for carryover contamination after a standard run.

10.1.2 An acceptable blank must meet the internal standard criteria stated in Section 8.10.1 and the surrogate recoveries found in Table 4. The concentration of the target compounds in the blank must be less than the method reporting limit for each target compound (see Table 7).

10.1.3 If a blank exceeds these criteria, corrective measures must be taken immediately, beginning with immediately reanalyzing the blank. If all criteria are still not met, the supervisor is then consulted.

10.2 Laboratory Control Spike (LCS and LCSD)

10.2.1 The Laboratory Control Spike (LCS and LCSD) are internal laboratory QC samples designed to show the capability of our laboratory to be in compliance with the protocol. They consist of filtered tap water spiked with an LCS solution which contains all compounds found in the daily calibration standards at a 10ug/L level.

10.2.2 An acceptable LCS and LCSD must meet the internal standard criteria stated in Section 8.10.1 and the surrogate recoveries in Table 4. The percent recovery for each of the compounds in the LCS and LCSD should be within recovery limits shown in the QC Method Reference "Blue Book". The LCS limits are set forth by our laboratory showing our internal control limits, therefore, operators should put forth every effort to meet these limits. Limits are derived using a control chart program as per the Control Chart SOP (SWL-GA-112).

10.2.3 If the compounds are outside QC limits, corrective action is needed immediately, beginning with rerunning the LCS to verify that it was prepared correctly and that it was not a bad purge. If reanalysis does not solve the problem, a new standard may need to be made.

10.3 Matrix Spikes - MS/MSDs will be analyzed at a rate of 1 MS/MSD per batch of 20 or fewer samples. A minimum of the five compounds (1,1-Dichloroethene, Benzene, Trichloroethene, Toluene, and Chlorobenzene) will be used; however all compounds can be spiked. The recovery limits will be the same as the LCS/LCSD and will be advisory since they are dependent upon the matrix.

11.0 WASTE DISPOSAL

11.1 Residual sample material and standards are discarded as directed by the Hazardous Waste Management SOP (SWL-GENHAZW, Rev. 3.0).

12.0 METHOD PERFORMANCE

12.1 The recovery limits for each of the compounds in the LCS and LCSD are shown in the QC Method Reference "Blue Book".

12.2 The recovery limits for each of the compounds in the MS and MSD are shown in the QC Method Reference "Blue Book".

12.3 MDL'S for this method are reported in the Laboratory's *"Red Book"*.

13.0 REFERENCES

13.1 Method SW846-8260A (Revision 1 – September, 1994)

13.2 Control Chart SOP (SWL-GA-112, Rev. 2.0, 5/15/97)

13.3 Definition and Determination of Detection Limits SOP (SWL-GA-113, Rev. 3.0, 6/17/97)

13.4 Hazardous Waste Management SOP (SWL-GENHAZW, Rev. 3.0)

14.0 DEFINITIONS

14.1 BFB: Bromofluorobenzene

14.2 Calibration: Establishment of an analytical curve based on response factors of known standards.

14.3 Calibration Standards: Series of known standard solutions used by the analyst for calibration of the instrument (i.e.: preparation of the analytical curve).

14.4 GC/MS: Gas Chromatography/Mass Spectrometry

14.5 Laboratory Control Spike (LCS): A known matrix spiked with compounds representative of the target analytes; Used to document laboratory performance.

15.0 DATA ARCHIVAL

15.1 Instrument data is stored in two separate locations before being archived to ensure data does not become erased or corrupt. Data is stored on both the instrument's Chemstation and the Unix-based processing system. Data is archived from the Unix-based system using the "TAR" format onto 60 m DAT tapes. No more than two months of data is archived onto the same tape. Once a tape contains two months of data, a tape directory is printed and bound for permanent storage. Each tape is labeled with the instrument ID and the date range of data contained on the tape.

16.0 METHOD DETECTION LIMITS (MDL'S)

16.1 MDL's are analyzed annually for 25mL water methods. Seven aliquots are analyzed at a concentration of 1 ug/L and their data is recorded in a spreadsheet format for easy review.

16.2 MDL's are verified to be below our reporting limit.

16.3 MDL'S are reported in the Laboratory's *"Red Book"*.

Table 1

Characteristic Ions for Surrogates and Internal Standards for Volatile Organic Compounds

Surrogate or Internal Standard	Primary Characteristic Ion	Secondary Characteristic Ion(s)	Surrogate or Internal Standard	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Dibromofluoromethane	113		Pentafluorobenzene	168	
1,2-Dichloroethane- d_4	65	102	1,4-Difluorobenzene	114	
Toluene- d_8	98		Chlorobenzene-d_5	117	
4-Bromofluorobenzene	95	174,176	1,4-Dichlorobenzene-d_4	152	

Table 2

Characteristic Masses (m/z) for Purgeable Organic Compounds

Analyte	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Acetone	43	58
Acrolein	56	55
Acrylonitrile	53	52, 51
Benzene	78	—
Bromobenzene	156	77, 158
Bromochloromethane	128	49, 130
Bromodichloromethane	83	85, 127
Bromoform	173	175, 254
Bromomethane	94	96
2-Butanone	43	72, 57
n-Butylbenzene	91	92, 134
sec-Butylbenzene	105	134
ert-Butylbenzene	119	91, 134
rbon Disulfide	76	78
arbon Tetrachloride	117	119
Chlorobenzene	112	77, 114
Chloroethane	64	66
2-Chloroethylvinyl Ether	63	43, 43
Chloroform	83	85
Chloromethane	50	52
2-Chlorotoluene	91	126
4-Chlorotoluene	91	126
cis-1,3-Dichloropropene	75	77
1,2-Dibromo-3-Chloropropane	75	155, 157
Dibromochloromethane	129	127
1,2-Dibromoethane	107	109, 188
Dibromomethane	93	95, 174
1,2-Dichlorobenzene	146	111, 148
1,3-Dichlorobenzene	146	111, 148
1,4-Dichlorobenzene	146	111, 148
Dichlorodifluoromethane	85	87
1,1-Dichloroethane	63	65, 83
1,2-Dichloroethane	62	98
-Dichloroethene	96	61, 63
1,2-Dichloroethene	96	61, 98
Trans-1,2-Dichloroethene	96	61, 98
1,2-Dichloropropane	63	112
1,3-Dichloropropane	76	78
2,2-Dichloropropane	77	97
1,1-Dichloropropene	75	110, 77
Ethylbenzene	91	106
2-Hexanone	43	58,57
Hexachlorbutadiene	225	223, 227
Isopropylbenzene	105	120
p-Isopropyltoluene	119	134, 91
Methylene Chloride	84	86, 49
4-Methyl-2-Pentanone	43	58
Naphthalene	129	—
n-Propylbenzne	91	120
Styrene	104	78
1,1,1,2-Tetrachloroethane	131	133, 119
1,1,2,2-Tetrachloroethane	83	131, 85
Tetrachloroethene	166	168, 129
Toluene	92	91
Trans-1,3-Dichlorpropene	75	77
1,2,3-Trichlorobenzene	180	182, 145
1,2,4-Trichlorobenzene	180	182, 145
1,1,1-Trichloroethane	97	99, 61
1,1,2-Trichlorethane	83	97, 85
Trichloethene	95	130, 132
Trichlorofluoromethane	101	103
1,2,3-Trichloropropane	75	77
1,2,4-Trimethylbenzene	105	120
1,3,5-Trimethylbenzene	105	120
Vinyl Chloride	62	64
o-Xylene	106	91
m,p-Xylene	106	91
Vinyl Acetate	43	86

Table 3

BFB Key Ions and Abundance Criteria

Mass	Ion Abundance Criteria
50	15.0 to 40% of mass 95
75	30 to 60% of mass 95
95	Base Peak, 100% Relative Abundance
96	5 to 9% of mass 95
173	<2% of mass 174
174	>50% of mass 95
175	5 to 9% of mass 174
176	>95% but <101% of mass 174
177	5 to 9% of mass 176

Table 4

Surrogate Recovery Limits*

Surrogate Compound	Water
Dibromofluoromethane	86-118
1,2-Dichloroethane- d_4	80-120
Toluene-d_8	88-110
4-Bromofluorobenzene	86-115

*Based upon method limits

Table 5

Volatile Internal Standards with Corresponding Analytes Assigned for Quantitation

Pentafluorobenzene	Chlorobenzene-d5	1,4-Difluorobenzene	1,4-Dichlorobenzene-D4
Acetone	Bromoform	Benzene	Bromobenzene
Acrolein	Chlorobenzene	Bromodichloromethane	n-Butylbenzene
Acrylonitrile	1,3-Dichloropropane	Bromofluorobenzene (Surrogate)	sec-Butylbenzene
Bromochloromethane	Ethylbenzene	Carbon Tetrachloride	tert-Butylbenzene
Bromomethane	2-Hexanone	2-Chloroethylvinylether	2-Chlorotoluene
2-Butanone	Styrene	1,2-Dibromoethane	4-Chlorotoluene
Carbon Disulfide	1,1,1,2-Tetrachloroethane	1,2-Dichloroethane	1,2-Dibromo-3-Chloropropane
Chloroethane	Tetrachloroethene	1,2-Dichloroethane- d_4 (Surrogate)	1,2-Dichlorobenzene
Chloroform	Dibromochloromethane	Dibromofluoromethane (Surrogate)	1,3-Dichlorobenzene
Chloromethane	m,p-Xylene	1,2-Dichloropropane	1,4-Dichlorobenzene
Dichlorodifluoromethane	o-Xylene	1,1-Dichloropropene	Hexachlorobutadiene
1,1-Dichloroethane		cis-1,3-Dichloropropene	Isopropyl benzene
1,1-dichloroethene		trans-1,3-Dichloropropene	p-Isoproyltoluene
cis-1,2-Dichloroethene		4-Methyl-2-Pentanone	Naphthalene
trans-1,2-Dichloroethene		Toluene	n-Propylbenzene
2,2-Dichloropropane		Toluene-d_8 (Surrogate)	1,1,2,2-Tetrachloroethane
Iodomethane		1,1,2-Trichloroethane	1,2,3-Trichlorobenzene
Methylene Chloride		Trichloroethene	1,2,4-Trichlorobenzene
1,1,1-Trichloroethane			1,2,3-Trichloropropane
Trichlorofluoromethane			1,2,4-Trimethylbenzene
Vinyl acetate			1,3,5-Trimethylbenzene
Vinyl chloride			

Table 6

Volatile Calibration Check Compounds (CCC)

- 1,1-Dichloroethene
- 1,2-Dichloropropane
- Ethylbenzene
- Chloroform
- Toluene
- Vinyl Chloride

Initial Calibration: 1,4,10,20,40ppb (25mL purge)
Max%RSD: 30%
Max%Drift: 20%
Calibration Verification: 10ug/L (25mL purge)

Volatile System Performance Check Compounds (SPCC)

- Chloromethane
- 1,1-Dichloroethane
- Bromoform
- 1,1,2,2-Tetrachloroethane
- Chlorobenzene

Minimum Relative Response Factor (RRF)
25mL Purge RRF: 0.100 for Chloroethane
1,1-Dichloroethane
0.300 for Chlorobenzene

Table 7

Compound List and Method Reporting Limits

Volatile Compound	CAS#	5mLWater or 5g Soil*	Volatile Compound	CAS#	5mL Water or 5g Soil*
Acetone	67-64-1	5	Trans-1,2-Dichloroethene	156-60-5	1
Acrolein	107-02-8	50	1,2-Dichloropropane	78-87-5	1
Acrylonitrile	107-13-1	50	cis-1,3-Dichloropropene	10061-01-5	1
Benzene	71-43-2	1	trans-1,3-Dichloropropene	10061-02-6	1
Bromobenzene	108-86-1	1	1,3-Dichloropropane	142-28-9	1
Bromochloromethane	74-97-5	1	2,2-Dichloropropane	594-20-7	1
Bromodichloromethane	75-27-4	1	1,1-Dichloropropene	63-58-6	1
Bromoform	75-25-2	1	Ethylbenzene	100-41-4	1
Bromomethane	71-83-9	1	2-Hexanone	591-78-6	5
2-Butanone	78-93-3	5	Hexachlorobutadiene	87-68-3	1
Carbon Disulfide	75-15-0	1	Isopropylbenzene	92-82-8	1
n-Butylbenzene	104-51-8	1	p-Isopropyltoluene	99-87-6	1
sec-Butylbenzene	135-98-8	1	Methylene chloride	75-09-2	1
tert-Butylbenzene	98-06-6	1	4-Methyl-2-Pentanone	108-10-1	5
Carbon Tetrachloride	56-23-5	1	Naphthalene	91-20-3	1
Chlorobenzene	108-90-7	1	n-Propylbenzene	103-65-1	1
Chloroethane	75-00-3	1	Sytrene	100-42-5	1
Chloroform	67-66-3	1	1,1,1,2-Tetrachloroethane	630-20-6	1
2-Chloroethylvinyl Ether	110-75-8	1	1,1,2,2-Tetrachloroethane	79-34-5	1
Chloromethane	74-87-3	1	Tetrachloroethene	127-18-1	1
2-Chlorotoluene	95-49-8	1	Toluene	108-88-3	1
4-Chlorotoluene	106-43-4	1	1,2,3-trichlorobenzene	87-61-6	1
Dibromochloromethane	124-48-1	1	1,2,4-Trichlorobenzene	120-82-1	1
1,2-Dibromo-3-Chloropropane	92-12-8	1	1,1,1-Trichloroethane	71-55-6	1
1,2-Dibromoethane	106-93-4	1	1,1,2-Trichloroethane	79-00-5	1
Dibromomethane	74-94-3	1	Trichloroethene	79-01-6	1
1,2-Dichlorobenzene	95-50-1	1	Trichlorofluoromethane	75-69-4	1
1,3-Dichlorobenzene	541-73-1	1	1,2,3-Trichloropropane	96-18-4	1
1,4-Dichlorobenzene	106-46-7	1	1,2,4-Trimethylbenzene	95-63-6	1
Dichlorodifluoromethane	75-71-8	1	1,3,5-Trimethylbenzene	108-67-8	1
1,1-Dichloroethane	75-34-3	1	Vinyl Chloride	75-01-4	1
1,2-Dichloroethane	107-06-2	1	o-Xylene	95-47-6	1
1,1-Dichloroethene	75-35-4	1	m,p-Xylene	13-302-07	1
cis-1,2-Dichloroethene	156-59-2	1	Vinyl Acetate	108-05-4	1

NOTE: Concentration Units: Water=µg/L
*Reporting Limits derived from method.

Table 8

Standards Preparation—25mL Purge

Compound or Standard Mix	Begin Conc. (µg/mL)	Aliquot (µL)	Total Vol. (mL)	Final Conc. (µg/mL)
Internal Standard				
NSI:				
Internal Standard Mix	2500	200	20	25
Surrogate:				
NSI:				
Surrogate Mix	2500	200	20	25
VSTD025				
NSI:				
Mix A	2000	125	10	25
Mix C	2000	125	10	25
Mix L	2000	125	10	25
Mix M	2000	125	10	25
Carbon Disulfide	5000	50	10	25
Vinyl Acetate	5000	50	10	25
Ketones Mix	1000	125	10	25
Extra Compound Mix				
Chem-Service:				
2-Chloroethylvinyl Ether	10,000	25	10	25
Acrolein	10,000	250	10	250
Acrylonitrile	10,000	250	10	250
1-Chlorohexane	10,000	25	10	25
MTBE	10,000	25	10	25
Methyl Iodide	10,000	25	10	25
Trans-1,4-Dichloro-2-Butene	10,000	25	10	25
n-Hexane	10,000	25	10	25
Gases Mix				
NSI:				
Gases Mix	1000	125	5	25

Table 9

Response Factor and Concentration Calculation

$$RF = \frac{(A_x)\ (C_{is})}{(A_{is})\ (C_x)} \quad \text{and} \quad conc_{RF} = \frac{(A_x)\ (C_{is})}{(A_{is})\ (\overline{RF})} \quad \text{or} \quad conc_{LR} = \frac{(A_x) - b}{M} \quad \text{or} \quad conc_{quad} = a + bx + cx^2$$

Where:

A_x = Area of characteristic ion for the measured compound.
A_{is} = Area of the characteristic ion for the specified internal standard
C_{is} = Concentration of the internal standard (ng/µL)
C_{RF} = Concentration of the measured compound (ng/µL) using the average response factor
$\overline{RF}$ = Average of Five Initial RF's for a compound
C_{LR} = Concentration of the measured compound (ng/µL) using Linear Regression
C_{quad} = Concentration of the measured compound (ng/µL) using the Quadratic Equation

---

Surrogate Recovery: % Surrogate Recovery = $\frac{Qd}{Qa} \times 100\%$

Where:

Qd = Quantity determined by analysis
Qa = Quantity added to sample

Matrix Spike recovery and RPD:

Matrix Spike Recovery = $\frac{SSR - SR}{SA} \times 100$ Matrix Spike RPD = $\frac{|MSR - MSDR|}{(½)(MSR + MSDR)} \times 100$

Where:

SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

Laboratory Control Spike (LCS) Recovery: LCS Spike Recovery = $\frac{SSR - SR}{SA} \times 100$

Where:

SSR = Spiked Sample Result
SR = Sample Result
SA = Spike Added

Calibration Calculations:

$$\%RSD = \frac{SD}{\overline{RF}} \times 100\% \qquad \%Drift = \frac{(C_1 - C_c)}{C_1} \times 100$$

Where:

SD = Standard Deviation
$\overline{RF}$ = Average of Five Initial RF's for a compound
RSD = Relative Standard Deviation

Where:

C_1 = Calibration Check Compound Standard Concentration
C_c = Measured concentration using selected quantitation method

VOLATILES GC/MS pH LOG

CASE _____ SDG# _____

LABORATORY ID	CLIENT SAMPLE ID	pH	ANALYST	DATE TESTED	DATE P.O. NOTIFIED*

COMMENTS: _____

- Date Project Officer Notified: Field is filled out when samples are not acidified and Project Officer is notified of situation

Figure 1. pH Log

VOLATILES GC/MS RUN LOG

VOLATILES GC/MS DEPARTMENT

SWOK / AATS

MS Std. #
IS Std. #
Surr Std. #
TUNE FILE ID:
METHOD FILES
INSTRUMENT:
DATE
CB/ID FILE ID:
ARCHIVE TAPE #:

Time	File	SWOK/AATS Sample ID	Client ID	Vol.	IS Area	Surr. Rec.	Lin. Chk.	Comments	ALS POS	OK/ RR

149

Analyst Signature

SWOK/AATS • 1700 West Albany • Broken Arrow, Ok 74012 [VGM008-0296-03]

Figure 2. GC/MS Run Log

SOUTHWEST LABORATORY OF OKLAHOMA, INC. *and*
AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.

TM

Standard Operating Procedure

Solid Sample Preparation

Document No.: SWL-RD-115

Rev No. / Date: Rev. 2.2 — 07/20/00

OFFICIAL COPY RED STAMP INDICATES ORIGINAL

APPROVALS	DATES
Procedure Prepared By	Date 7/20/00
Program Manager or Section Supervisor	Date 7/20/00
Laboratory Safety Officer	Date 7/21/2000
Laboratory QA/QC Officer	Date 7/20/00

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SOLID SAMPLE PREPARATION

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1.0 SCOPE AND APPLICATION

1.1 This procedure details the steps necessary to prepare soil/solid samples for the digestion operation (SWL-RD-126) or for direct use as needed. Following this procedure will ensure that soils are blended. dried and of a sufficiently small particle size to be effectively utilized through the remaining analytical processes required to satisfy the parameters requested.

1.2 This procedure is performed when specifically client requested.

2.0 METHOD SUMMARY

Each sample is dried in a drying oven and then pulverized to a very small grain size in a ball mill. After milling the dried sample, each sample is sieved to collect the small particulate and stored for future use as needed.

3.0 HEALTH AND SAFETY

3.1 Perform sieving samples and putting samples into or removing samples from the ball mill cans in well-ventilated areas only to avoid inhaling the dust. See *Laboratory Protection Plan*, SWL-GA-130.

3.2 See Standard Operating Procedure *Laboratory Safety Plan,* SWL-GA-111 and *Respiratory Safety,* SWL-RD-141.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING & STORAGE

Not applicable.

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

Be sure to thoroughly clean area between each sample and after each group of samples to avoid cross contamination or the spread of contamination.

6.0 EQUIPMENT/APPARATUS

6.1 Ball mill

6.2 One-quart paint cans.

6.3 Three 1-inch diameter stainless steel bearings per sample.

6.4 Drying oven capable of maintaining 105 ± 5 degrees C.

6.5 200 mesh sieve

6.6 Capable centrifuge tubes (50 milliliter).

6.7 Glass drying dish, (Pyrex or equivalent)

7.0 REAGENTS

Radiac Wash or equivalent.

8.0 PROCEDURE

8.1 The radiochemist/analyst who is experienced in standard wet preparation methods and who is trained in the use of this procedure shall be permitted to perform this procedure.

8.2 Pre-weigh a numbered drying dish. Enter the tare weight into the dry/ash logbook.

8.3 Place the entire sample up to 50% of the ball mill canister volume (excluding sufficient volume for tritium where required) into the drying dish and re-weigh to obtain the gross wet weight. Subtract the tare weight from the gross wet weight to obtain the net wet weight. Record both the gross weight and net weight in the dry/ash logbook. (note: If the total original sample volume is greater than 50% of the ball mill canister volume, then take an representative aliquot of the original sample that will fill the canister to make 50% of its total available volume.)

8.4 Dry the entire sample at least 12 hours at approximately 105° ± 5° C. After the drying period, examine the material to see if it is dry by probing with a cleaned glass rod. If the material is not dry, then continue the drying process until completely dry.

8.5 Remove the sample from the drying oven, allow it to cool and re-weigh. Subtracting the tare weight from, record the net dry weight in the dry/ash logbook. (The percent moisture can be obtained utilizing the net wet and net dry weight figures).

8.6 Place the entire sample in a new one-quart size paint can along with three, one-inch diameter stainless steel bearings and secure the lid. A larger paint can may be used if a larger sample is needed.

8.7 Place the paint can on the ball milling device and rotate for at least 45 minutes to 1 hour.

8.8 Remove the can from the ball mill and open it under a hood.

8.9 Sieve the sample through a 200-mesh sieve collecting the sieved material in a new capable centrifuge tube that is appropriately labeled. Each labeled sample shall clearly indicate that the sample has been sieved. If excess sieved material remains after filling the centrifuge tube, return it to the original sample container along with the material that did not pass through the sieve. The ball bearings are removed at this time and cleaned prior to reuse.

8.10 Wash both the sieve and the ball bearings with a Radiac Wash or equivalent solution followed by a D.I. water rinse prior to reuse. Towel-dry the ball bearings and oven dry both until dry for approximately 10 minutes.

8.11 For samples that need to be acid digested, proceed to standard operating procedure, SWL-RD-126, "Acid Digestion for Soils and Air Filters" making sure to take a representative subsample. Refer to each individual SOP or client specific statements of work as needed.

8.12 Records of soil weights prepared are kept in the Dry/Ash logbook.

9.0 CALCULATIONS

Not applicable.

10.0 QUALITY ASSURANCE/QUALITY CONTROL

See Standard Operating Procedure *Quality Control for Radiochemical Analyses*, SWL-RD-113.

11.0 WASTE DISPOSAL/POLLUTION PREVENTION

See Standard Operating Procedure *Hazardous Waste Management Plan*, SWL-GA-114.

12.0 METHOD PERFORMANCE

Not applicable.

13.0 REFERENCES

Not applicable.

14.0 DEFINITIONS

Not applicable.

15.0 ATTACHMENTS

- Attachment 1: Dry/Ash LogBook.

ATTACHMENT 1
Dry/Ash Log Book

Dry/Ash Log Book , SWL-RD115

1

DRY/ASH LOG BOOK (115-ATT1.DOC) SWL-RD-115, Rev.:____ Book # RD115-PRP-__1__

Verified ____

Date	Analyst	Dish #	Smple. ID	Dish Weight	Dish + Samp. Wet Weight	Smple. Wet Wt.	Dish + Samp. Dry Weight	Smple. Dry Wt.	% Dry

Southwest Laboratory of Oklahoma, Broken Arrow, OK

SOUTHWEST LABORATORY OF OKLAHOMA, INC. *and*
AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.

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Standard Operating Procedure

Gamma Spectrometer Sample Preparation

Document No.: SWL-RD-118

Rev No. / Date: Rev. No.: 1.2 — 03/29/99

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APPROVALS	DATES
Kent Smith Procedure Prepared By	3/29/99 Date
Kent Smith Program Manager or Section Supervisor	3/29/99 Date
Chuck Hoover Laboratory Safety Officer	4/2/99 Date
Chuck Hoover Laboratory QA/QC Officer	4/2/99 Date

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GAMMA SPECTROMETER SAMPLE PREPARATION

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1.0 SCOPE AND APPLICATION

The purpose of this procedure is to provide a step by step instruction for the preparation of samples for gamma spectrometry analysis.

2.0 METHOD SUMMARY

Samples are aliquoted and prepared in sample containers (geometry) to match calibration geometries to be analyzed for gamma emitting isotopes.

3.0 HEALTH AND SAFETY

3.1 Refer to the current version of the *"Laboratory Safety Plan"* SWL-GA-111, and SWL-RD-141 *"Radiation Safety"*.

3.2 Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Wearing gloves, eye protection, and a laboratory coat during extraction and analysis is advisable and should provide the proper protection.

3.3 The following chemicals have the potential to be highly toxic or hazardous, consult MSDS: All Acids.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

Not applicable.

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

Not applicable.

6.0 EQUIPMENT/APPARATUS

6.1 Top loading balance

6.2 Various sample containers.

6.3 Marinelli beakers.

7.0 REAGENTS

Not applicable.

8.0 PROCEDURE

8.1 Liquid Sample Preparation

8.1.1 All liquid samples are measured out in graduated cylinders and poured into appropriate sized containers and sealed with appropriate lids.

NOTE: Containers used for each sample shall match those used for instrument calibration. Consult the instrument room personnel for appropriate containers to use and match accordingly.

8.1.2 Seal lip of container with tape to avoid evaporation.

8.2 Solids Preparation:

Solids are placed into appropriate containers that correspond to a known geometry for gamma spectrometry analysis. The container is first tare weight measured and then the sample is added to determine net sample weight. Dry weight of sample is determined by using previously determined dry/wet ratio of samples multiplied by net sample weight to get dry weight of sample used. This dry weight value is used for sample volume size in the sample activity determinations.

8.3 Record preparation activities in the Gamma Spec Prep. Log.

8.4 Submit samples for Gamma Spectroscopy.

9.0 CALCULATIONS

Not applicable.

10.0 QUALITY ASSURANCE/QUALITY CONTROL

See Standard Operating Procedure "*Quality Control for Radiochemical Analyses*", SWL-RD-113.

11.0 WASTE DISPOSAL

See Standard Operating Procedure "*Hazardous Waste Management Plan*", SWL-GA-114.

12.0 METHOD PERFORMANCE

Not applicable.

13.0 REFERENCES

SWLO Standard Operating Procedures, G-Series.

14.0 DEFINITIONS

Not applicable.

15.0 ATTACHMENTS

15.1 Attachment 1: Gamma Spec. Prep Form

ATTACHMENT 1

Gamma Spec. Prep Form

Gamma Spec. Prep, SWL-RD118

GAMMA SPEC. PREP FORM (118-ATT1.DOC) Rev.#:_____ Book # RD118-PRP-__1__

Verified ___

Date	Analyst	Sample ID	Matrix	Sample Size	Container

SOUTHWEST LABORATORY OF OKLAHOMA, INC. and
AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.

Standard Operating Procedure

Isotopic Thorium and Neptunium

Document No.: SWL-RD-120

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Program Manager of Section Supervisor	Date 8/22/00
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ISOTOPIC THORIUM AND NEPTUNIUM

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1.0 SCOPE AND APPLICATION

The purpose of this procedure is to provide a step by step direction for the separation and subsequent isotopic measurement of thorium and neptunium by alpha spectroscopy using Eichrom Ind., Inc. "TEVA" ion exchange resins. This procedure applies to primarily aqueous and soil/solid samples but can be used for other matrixes after digestion procedures have been followed.

2.0 METHOD SUMMARY

2.1 This procedure provides separation of thorium and neptunium elements from other interfering analytes by ion-exchange techniques. Standard Operating Procedure SWL-RD-107 "*Isotopic Thorium*" can be used alternatively for the thorium chemical separation in place of this procedure.

2.2 Aqueous (or digestates of solid samples) are acidified and coprecipitated on Calcium Phosphate thus separating elements of interest, (thorium and neptunium), from other radioactive elements. This precipitate is dissolved into a solution which is ran through various ion exchange resins in various acid matrixes to chemically separate each elemental fraction into the thorium and neptunium fractions and submitted for alpha spectroscopy. The final samples are coprecipitated on cerium fluoride.

2.3 This procedure is based upon method EPA 908.0 and EPA 907.0. Due to research over the years, newer technology has enabled improvement of the original methods in determining isotopic activities for alpha emitting radionuclides. These improvements are also necessary due to the fact that lower detection limits are required. The original intent of the method was to determine the above isotopically. This procedure accomplishes that by using commercial ion exchange resins and alpha spectroscopy . These "newer" resins use differing initial precipitations that are compatible with their particular ionic mechanism for elemental separations from interfering alpha emitting radionuclides.

EPA 907.0 primarily uses various stages of precipitation and organic extractions to separate the elements of concern, and then uses analysis by alpha scintillation to determine isotopic abundances.

EPA 908.0 primarily uses various stages of precipitation and ion exchange resins to separate the elements of concern, and then uses analysis by gas proportional counting to determine gross activities.

The changes described herein to the original methods has not changed the intended end product of the method.

3.0 HEALTH AND SAFETY

3.1 Refer to the current version of the "*Laboratory Safety Plan*" SWL-GA-111 and SWL-RD-141, "*Radiation Safety*".

3.2 Each chemical should be regarded as a potential health hazard and exposure should be as

low as reasonably achievable. Wearing gloves, eye protection, and a laboratory coat during extraction and analysis is advisable and should provide the proper protection.

3.3 The following chemicals have the potential to be highly toxic or hazardous, consult MSDS: See Section 7.0 and become familiar with each chemical used.

Note: Hydrofluoric acid is used in this procedure, and thus Hydrofluoric acid neutralizing agent should always be available for use if needed.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Not Applicable.

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

Not Applicable.

6.0 EQUIPMENT / APPARATUS

6.1 Analytical Balance – 0.0001g sensitivity

6.2 Centrifuge

6.3 Centrifuge tubes

6.4 Column Racks

6.5 Filter apparatus – Gelman filter apparatus with polycarbonate base and poly screen, polysulfide funnel.

6.6 Filter - .45 micron

6.7 Gelman filter 25mm 0.1 micron (product number HT-100) or equivalent

6.8 Fume hood

6.9 Hotplate

6.10 1-liter beakers

6.11 Teflon stir rods

6.12 100ml beakers

6.13 250ml beakers (waste collection)

7.0 REAGENTS

7.1 ASTM type II, De-ionized Water.

7.2 Ammonium hydrogen phosphate (3.2 M)-Dissolve 104g of $(NH_4)2HPO_4$ in 200ml of

water, heat gently to dissolve, and dilute to 250ml with water.

7.3 Ammonium hydroxide- concentrated (sp gr 0.9)

7.4 Appropriate tracers or standards

7.5 Ascorbic acid

7.6 Calcium nitrate (1.25M)- Dissolve 51g of $Ca(NO_3)_2$ in 100ml of water and dilute to 250mls with water

7.7 Cerium carrier-Dissolve 0.155g cerium (III) nitrate hexahydrate in 50ml water and dilute to 100mls with water

7.8 Ethanol, 80%-Add 80ml ethanol to 20ml water.

7.9 Ferrous sulfamate solution (0.6M)-Add 57g of NH_2SO_3H to 150ml of water, heat to 70° C, add 7g of iron powder (< 100 mesh size) in small increments until dissolved, filter (Whatman #4 suggested), transfer to flask and dilute to 200ml with water. ***Prepare fresh weekly ***.

7.10 Hydrochloric acid (9 M)- Add 750ml of concentrated HCl (sp gr 1.19) to 100ml of water and dilute to 1 L with water.

7.11 Hydrochloric acid (12M)-concentrated HCl (sp gr 1.19).

7.12 Hydrochloric acid (2 M) – Add 167 mls of concentrated HCL (sp gr 1.19) to 500mls of water and dilute to 1 liter with water.

7.13 Hydrofluoric acid (28 M) -concentrated hydrofluoric acid (sp gr 1.2).

7.14 Nitric acid (15.7M)-concentrated HNO_3 (sp gr 1.42)

7.15 Nitric acid (2.5 M) – ferrous sulfamate solution (0.1 M) – Add 159mls of concentrated HNO_3 (sp gr 1.42) and add 166mls of .6 M ferrous sulfamate solution to 500mls of water and dilute to 1 L with water. *** Prepare fresh daily ***.

7.16 Nitric acid (0.02M)- hydrofluoric acid (0.02 M) - Add 0.64mL of concentrated HNO_3 (sp gr 1.42) and 0.36mL of concentrated HF to 400mL of water and dilute to 500mL with water.

7.17 Nitric acid solution (2.5M) - Add 159mL of concentrated HNO_3 (sp gr 1.42) to 800mL of water and dilute to 1L with water.

7.18 Nitric acid solution (3 M) - Add 191ml of concentrated HNO_3 (sp gr 1.42) to 700ml of water and dilute to 1 L water.

7.19 Nitric acid solution (6 M) - Add 380ml of concentrated HNO_3 (sp gr 1.42) to 500ml of water and dilute to 1 L water.

7.20 Nitric acid solution (3M)- Aluminum nitrate (1.0M) solution - Add 191ml of

concentrated HNO_3 (sp gr 1.42) to 700ml of water. Dissolve 213g anhydrous aluminum nitrate and dilute to 1 L with water. (3M HNO_3 - 1M $Al(HNO_3)_3$).

7.21 Hydrogen Peroxide. (30 %)

7.22 Phenolphthalein - Indicator-Dissolve 1g phenolphthalein in 50mL ethyl alcohol and dilute with 50mL of water.

7.23 TEVA Resin column (Eichrom Ind., Inc.). 100-150um resin or small particle size (50-100um) in appropriate plastic column.

8.0 PROCEDURE

8.1 Place required aliquot of sample to achieve desired detection limits in a glass beaker.

Add: ~5 mls of 12N Hydrochloric Acid to acidify the sample.

Standards and Tracers. Use Th-229 or Th-234 as the tracer depending upon client requirements. (If using Th-234 as the tracer see SWL-RD-111 "*Thorium-234 Preparation*").

Do not add tracers to soil samples that have been digested under SWL-RD-126 "*Acid Digestion for Soils and Air Filters*" for alpha spectroscopy analysis. Tracers should have been added prior to digestion. Assure that tracers were added to these samples prior to digestion before continuing with the next step. If tracers were not added prior to digestion, report the deficiency to the laboratory supervisor and arrange to have the sample digestion repeated.

8.2 Add 0.5ml of 1.25 M $Ca(HNO_3)_2$ to each beaker.

8.3 Heat samples until boiling.

8.4 Once the samples boil, take the watch glass off of the beaker and turn the heat down to medium.

8.5 Add 2-3 drops of phenolphalein indicator.

8.6 Add 200uL of 3.2M $(NH_4)2HPO_4$ solution.

8.7 Add enough concentrated NH_4OH with a squirt bottle to reach the phenolphthalein end point and form $Ca_3(PO_4)_2$ precipitate. NH_4OH should be added very slowly. Stir the solution with a glass or Teflon stir rod. Allow the sample to heat for another 20-30 minutes.

8.8 Allow precipitate to settle until solution can be decanted (30 minutes to 2 hours).

8.9 Decant supernatant and discard to waste.

8.10 Transfer the remaining precipitate to a centrifuge tube and centrifuge the precipitate for approximately 5 minutes at 2000rpm. Decant supernatant and discard to waste.

8.11 Wash the precipitate with an amount of D.I. water approximately twice the volume of the precipitate. Mix well on a vortex mixer. Centrifuge for 5-10 minutes. Discard the supernatant to waste.

8.12 If an ammonia odor persists repeat the last water wash step above.

8.13 Dissolve precipitate in approximately 5ml concentrated nitric acid. Transfer solution back to the original beaker. Rinse centrifuge tube with 2-3mls of concentrated nitric acid and transfer to beaker. Evaporate solution to dryness.

8.14 Dissolve each precipitate with 10mls of 3.0 M HNO_3 - 1.0 M $Al(HNO_3)_3$.

Note: An additional 5mls may be necessary if the volume of precipitate is large.

8.15 Add 2.0mls of 0.6 M ferrous sulfamate to each beaker. Swirl to mix.

Note: If the additional 5mls was used to dissolve the sample from the step above, then add 1 more milliliter of 0.6 M ferrous sulfamate.

8.16 Add approximately 100 mg of ascorbic acid. Swirl to mix. Wait for 2-3 minutes.

Note: If particles are observed suspended in solution, centrifuge the sample.

The supernatant will be transferred to the TEVA column below. The precipitate will be discarded.

8.17 For each sample dissolved, place a TEVA Resin column in the column rack.

8.18 Place a waste beaker below each column, remove the bottom plug from each column and allow to drain if necessary.

8.19 Pipet 5mls of 3M HNO_3 into each column to condition resin and allow to drain as waste.

8.20 Transfer each redissolved sample into the appropriate TEVA Resin column by pouring or by using a plastic transfer pipet and allow to drain as waste.

8.21 Add 5mls of 2.5 M HNO_3 - 0.1 M ferrous sulfamate to rinse each beaker and transfer each solution into the appropriate TEVA Resin column. Discard eluate as waste.

Note: Pu, U and Am are removed with the loaded solution and 2.5 M HNO_3 - 0.1 M ferrous sulfamate rinses.

8.22 Add 30mls of 2.5 M HNO_3 - 0.1 M ferrous sulfamate into each column. Discard eluate as waste.

8.23 Add 5ml of 2.5 M HNO_3 to each column. Discard eluate as waste.

8.24 Place a **clean**, labeled 50ml beaker below each column.

8.25 Pipet 20ml of 9M HCL into each column and **collect eluate**. Pipet 5ml of 6M HCL in each column and continue to collect elute.

8.26 This beaker will contain the **thorium fraction**. Label this beaker as TH for the thorium

Typist: LLR

fraction.

8.27 If collecting the neptunium fraction place a **clean**, labeled 50ml beaker below each column.

8.28 Pipet 10ml of 0.02 M HNO_3 - 0.02 M HF into each column to strip the neptunium. Collect eluate. Label this beaker as NP for the **neptunium fraction**.

8.29 To each **thorium** and **neptunium** fraction beaker, cook down to just dryness.

8.30 Dissolve with 5mls conc. HNO_3 and 5 mls (30%) H_2O_2, cook down to just dryness.

8.31 Repeat step above.

8.32 Dissolve with 10mls 2M HCL, quantitatively transfer to plastic centrifuge tube rinsing beaker with 2 to 3 additional mls of 2M HCL into the centrifuge tube.

8.33 Add 0.2ml of cerium carrier to each centrifuge tube.

8.34 Add 1.0ml of conc. HF to each tube. Swirl to mix. Let the solutions sit for at least 30 minutes before filtering.

8.35 Set up a .1 micron 25mm filter, glassy side down on a Gelman filter apparatus.

8.36 Add 3-5mls of 80% ethanol to each filter, applying vacuum and ensuring there are no leaks along the sides. Add 2-3mls of deionized water to each filter.

8.37 Filter the sample and rinse 50ml centrifuge tube with 5mls water, transferring this rinse to the filter apparatus.

8.38 Wash each filter with 3-5mls of ethanol.

8.39 Remove filters, mount on appropriate disks.

8.40 Submit each filter for analysis by Alpha Spectroscopy, and fill out calculation pages and submit for calculations.

9.0 CALCULATIONS

9.1 Calculate each isotope of thorium and neptunium activity, A, in picocuries per volume used in "V" below as follows:

$$A = \frac{\frac{SC}{T} \quad \frac{BC}{T}}{\frac{IC/T}{Idpm} \times V \times 2.22}$$

Where:

SC = sample counts
BC = background counts
T = time in minutes
V = sample volume
IC = internal tracer counts
Idpm = internal tracer dpm
2.22 = conversion factor from dpm to pCi.

10.0 QUALITY ASSURANCE / QUALITY CONTROL

See Standard Operating Procedure "*Quality Control for Radiochemical Analyses*", SWL-RD-113.

11.0 WASTE DISPOSAL/POLLUTION PREVENTION

See Standard Operating Procedure "*Hazardous Waste Management Plan*". SWL-GA-114.

12.0 METHOD PERFORMANCE

Not Applicable.

13.0 REFERENCES

13.1 SWLO Standard Operating Procedures, G-series, and R-series as indicated.

13.2 Prescribed Procedures for Measurement of Radioactivity in Drinking Water, EPA-600/4-80-032, August, 1980. EPA. Methods 907 and 908 .

13.3 EMSL-LV-0539-17

13.4 Eichrom Industries, Inc. Analytical Procedures, ACW08, rev.1.4

13.5 SWL-RD-111, "Thorium 234 Preparation"

14.0 DEFINITIONS

Not Applicable.

15.0 ATTACHMENTS

Attachment 1: Isotopic Th/Np Prep Form

ATTACHMENT 1
Isotopic Th/Np Prep Form

Isotopic Th/Np Prep. SWL-RD120

ISOTOPIC TH/NP PREP FORM (120-ATT2.DOC) SOP REV.# ____ SWLC Book # RD120-PRP-____

Prep Date: _____ Analyst ___ Batch ID. _____ Verified ___

Sample Number	Volume	Sample Matrix	Column Number	Notes

Standard ID/ Th Activity : _____

Amount used : _____

Thorium Tracer ID/ Activity : _____

Amount Used : _____

Standard ID/ Np Activity : _____

Amount used : _____

Np Tracer ID/ Activity : _____

Amount Used: _____

SOUTHWEST LABORATORY OF OKLAHOMA, INC. *and*
AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.

Standard Operating Procedure

Gamma Spectrometer Operation/Calibration

Document No.: SWL-RD-125
Rev No. / Date: Rev. No.: 1.0 — 05/02/01

APPROVALS	DATES
Procedure Prepared By	Date
Program Manager or Section Supervisor	Date
Laboratory Safety Officer	Date
Laboratory QA/QC Officer	Date

(Effective Date is 5 calendar days after the last signature above - QA/QC Officer)

Document Status

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GAMMA SPECTROMETER OPERATION/CALIBRATION

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1.0 SCOPE AND APPLICATION

The purpose of this procedure is to supply a list of instructions and explanations to the method of calibration and operation of the Extended Range Germanium Detector (hpGe) for identification of gamma emitting radionuclides. This procedure is in updated form of EPA 901.1.

2.0 METHOD SUMMARY

After sample preparation as per standard operating procedures SWL-RD-118, those samples which require gamma spectroscopy analysis are analyzed and printed.

3.0 HEALTH AND SAFETY

3.1 Standard safety and radiological precautions should be observed. See Standard Operating Procedure "*Laboratory Safety Plan*", SWL-GA-111.

3.2 Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Wearing gloves, eye protection, and a laboratory coat during extraction and analysis is advisable and should provide the proper protection.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

Not applicable.

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

5.1 Sample containers outside surface area shall be cleaned thoroughly before being submitted to the count room for counting. This is to prevent any contamination of detectors and shielding.

5.2 The detector should never have voltage applied to it until it has been cooled with liquid nitrogen. The pre-activate cool down period for this detector is 6 hours. Also, special care should be taken when the shielding is open to avoid objects from falling into the shield and possibly damaging the detector.

6.0 EQUIPMENT/APPARATUS

6.1 Tennelec TC-950, Bias power supply.

6.2 Tennelec TC-243, Amplifier.

6.3 Canberra hpGE gamma detector.

6.4 Oxford GammaTrac V1.00 Software.

7.0 REAGENTS

7.1 Liquid Nitrogen.

8.0 PROCEDURE

8.1 Introduction

The gamma spectroscopy system is calibrated to analyze certain energies of gamma emitting isotopes by using known gamma emitting isotopic energies that correspond to desired isotopes. Interpolation methods are used to determine unknowns based on knowns used in calibration. Each detector is calibrated first by determining a channel to energy calibration, and then by determining detector efficiencies. In addition to the above calibrations, background count information is acquired and used per detector. These calibrations are used to determine sample isotopic energies and count activities that are used in software calculations to arrive at resultant activity values as needed. Sample count durations vary depending upon desired detection limits as required per client request. Once the detectors have been calibrated, periodic checks are made to assure that samples are only analyzed on calibrated detectors.

8.2 System Configuration

This system consists of a Canberra Reverse electrode closed-end coaxial high purity Ge (hpGe) gamma detector fitted with a thin window Be endcap at a relative efficiency of 35%. The detector is fitted with a Canberra pre-amp that is connected to a Tennelec TC-243 amplifier. The detector is supplied bias via a Tennelec TC-950 power supply.

8.3 Detector Calibration (Energy/Channel, Efficiency)

8.3.1 The system will be calibrated to each specific sample geometry (counting container) that will need to be counted. These geometries will vary depending upon specific client needs and availability of sample size. Calibration records for each geometry will be stored in the system and will be used as appropriate for the samples that are being analyzed.

8.3.2 Prepare a multi-line (energy) calibration standard by taking a known NIST traceable multi-nuclide standard solution and dilute it up to the needed volume and place in the desired geometry. These geometries shall be recorded and traceable by following standard operating procedures for standard preparation (SWL-RD-124). This sample volume and container geometry shall match samples that will be analyzed. The multi-nuclide standard solution should include gamma energies that range throughout the 0 to 2000 keV energy range. Most commercially available standards include isotopes Cd-109 @ 88 keV through Y-88 @ 1836 with various isotope/energies combinations between. These isotopes will have half-lives that can be short as in the case of Cr-51 @ 320 keV (27.7 days) and as long as Cs-137 @ 662 keV (30.17 years). The calibration is reported in the data package and will include the listing of the nuclides that were calibrated and subsequently checked as calibration check sources. The nuclides which have shorter half-lives will not need to be accounted for when energy peaks become two small to quantify. By using the nuclides with longer half-lives in the check standard the system is effectively monitored for integrity of calibration through time. The system shall be calibrated with a minimum of 5 energies that are distributed throughout the 0 to 2000 keV energy range.

8.3.3 The first step in the calibration procedure is a two-point energy calibration. This is needed for the refined calibration from the standard that will be used in sample analyses later.

8.3.4 Place the calibration standard geometry on the detector making sure that the position of the calibration standard is the that will be used when counting samples in the future.

8.3.5 Tightly close the shield door.

8.3.6 Open the Oxford GammaTrac V1.00 Software.

8.3.7 The two point initial calibration will use two energy peaks initially. Set the sample count preset time to a period that is long enough to display energy peaks that will accumulate at least 10,000 net counts to be used in calibration by pressing F3 and entering the time in seconds. Start the system by clearing the existing spectrum by pressing CTRL-F2 and then pressing F1 to start count.

8.3.8 After the count has finished, move the cursor over the first peak. Press the A-key to bring up the Analyze Menu. Select Calibrate system and press ENTER. The calibration parameters will be displayed. Set the following: energy calibration units: **keV**, use energy in master library: **No**, standards filename: ***.std**, fitted efficiency function: **log cubic polynomial**. When the required has been entered, press ESC. This brings up the Calibrate system menu with energy Calibration highlighted. Press ENTER.

8.3.9 Establish a Region of Interest (ROI) around the peak by pressing F9 to the left of the desired peak and F10 to the right of the desired peak. Press Enter. The ROI will be processed for a peak-fit and a prompt for the energy will be displayed. Type in the appropriate energy and press ENTER. An outline of the peak-fit will be displayed around the peak and the data-box at the lower part of the screen will show what has been input. A prompt for the next input will be displayed in the upper box.

8.3.10 Using the PgUp-key, move the cursor to next peak to be used in this two point calibration.

8.3.11 Establish an ROI around this and press ENTER. Ignore the energy displayed in the prompt box and type the correct energy for this ROI and press ENTER.

8.3.12 As before, an outline of the peak-fit will be displayed around the peak with corresponding data in the box at the lower part of the screen. The two-point calibration is now complete. Press the ESC-key to return to the Calibrate system menu.

8.3.13 The curve for the Energy calibration with data points shown can now be displayed by moving the highlighted box down to Display energy curve and pressing ENTER. The linear equation of this curve will be shown in the upper box. Use the "ESC" key to return to the Calibrate system menu. In a similar manner, the curve and equation for the FWHM can be seen.

8.3.14 Finally, move the highlighted box to Quit and update files and press ENTER. The box at the top of the screen will now prompt a reply as to whether the energy calibration data is to be written on the filed spectrum. Answer NO. The two-point energy and FWHM calibration is now complete.

8.3.15 Now conduct the fully automatic calibration of energy, FWHM and detector efficiency simultaneously.

8.3.16 As before, press 'A' to bring up the analyze menu, select Calibrate system, press ENTER and respond with 'Y' to the calibration parameters menu since they are now all correct. The Calibrate system menu is then displayed.

8.3.17 Move the highlight to calibrate from Standard and press ENTER.

8.3.18 From the calibrate Standard menu, select Full Calibration and press ENTER.

8.3.19 The spectrum will now be automatically searched, the peaks corresponding to the energies of the standard fitted, and the calibration data computed. Enter the appropriate information for each peak just as before. When completed, a revised calibrate from Standard menu is presented with Inspect/edit calibration peaks highlighted. Press ENTER.

8.3.20 Equations will be fitted to the calibration data. When completed, the Calibrate system menu will reappear. The energy, FWHM or Efficiency curves may now be displayed for review. Each will show the actual data points.

8.3.21 Use the "ESC" key to return to the Calibrate system menu. Press Quit and update the files. Press ENTER. The prompt in the upper box will inquire about saving this new efficiency data (Y or N). Press 'Y'.

8.3.22 Capture the efficiency file by typing in a name desired and press ENTER. This file will be used when analyzing future sample spectrums.

8.3.23 Follow the steps in the "Spectrum Count / Analysis" section of the SOP and count a Background spectrum which will be used to subtract background peaks.

8.3.24 Check the calibration file by following the next section "Spectrum Analysis" and using this new calibration file. By using the actual spectrum used in calibration and the derived calibration formulas, will show comparisons to the known values of the calibration spectrum.

8.3.25 This system shall be re-calibrated at least monthly for each geometry needed.

8.4 Spectrum Count / Analysis

8.4.1 Place the item to be counted on the detector making sure that the position is identical to that of the calibration standard.

8.4.2 Tightly close the shield door.

8.4.3 Open the Oxford GammaTrac V1.00 Software.

8.4.4 Set the sample count preset time to a period that is long enough to achieve required detection limits by pressing F3 and entering the time in seconds. Start the system by clearing the existing spectrum by pressing CTRL-F2 and then pressing F1 to start count.

8.4.5 After the count have finished, save the spectrum to file by pressing F7 and entering desired file name.

8.4.6 Analyze spectrums by entering F6 to load spectrum file.

8.4.7 Enter spectrum specific information by ALT-S(etup) as follows. Sample ID : , Sample Quantity: , Sample Units:, Time of Sample Irradiation: (This time should remain blank if decay correction is not to be used), Calibration filename: (Enter the filename of the calibration file for this geometry, *.cal), Efficiency filename: (Enter the filename of the efficiency file for this geometry, *.eff).

8.4.8 All of the parameters required for operating the Gamma Trac analysis facilities are contained in binary parameters files. You may have as many different files as you wish, each containing different sets of parameters for different types of analysis. On start-up the program suite always loads the last parameter file. To change the parameters you must use "new parameters file" and specify the file name you wish to use. By specifying a file name which does not already exist, this option will allow you to create a new file for future use. All parameters may be changed from their defaults in the "edit analysis parameters" facility. The parameters are grouped into various sections (e.g. "peak search parameters" etc.) and upon selection a panel the parameters are displayed. Note that the name of the current parameters file is displayed in the title bar and the bottom of the panel summarizes which keys can be used for editing. Use the 'F1' key to provide information on how each parameter is used. These parameters will printout on the analysis report to be reported with the results.

8.4.9 Setup the parameters file with the following as default unless client specific requirements are different. Peak search sensitivity : 5.0, Start energy for search: 20.0 (kev), Stop energy for search: 2000.0 (kev), No. of background channels: 5. Background peak subtraction is (On/Off as desired). Isotope library: (as desired).til, Match tolerance: 3.00 (kev)

8.4.10 Launch the fully automated mode which searches the spectrum, locates and fits peaks and then proceeds with the desired output form by entering the ALT-A(nalyze) key. Upon completion of the output table, the program returns to the beginning and is ready for accumulation of another spectrum.

8.4.11 The current Peak search parameters table will be displayed. This provides a number of options to facilitate the search. To change these, press 'N' as the upper box directs.

8.4.12 The prompt first moves to the 'low energy cut-off'. This directs the search to begin at the energy shown(or input) and allows low energy noise and x-rays to be ignored. If a new value is input, press ENTER. If the value displayed is OK, move with the 'down arrow' to high energy cut-off. Here the value shown

truncates the search to omit a region which may contain little information of interest.

8.4.13 When the desired parameters are present, press the Esc-key. The upper box will allow the search to proceed with 'Y' or be aborted with 'N'. A 'Y' will result in the programmed search to commence. The progress of the search will be shown in the upper box. Upon completion of the search, the peak-fitting will proceed and again the progress here can be monitored in the upper box. When completed, a 'beep' will sound and the peak search menu will appear with View peak fit highlighted. Press ENTER. The program will continue with the entire procedure of analysis is executed with designated output.

8.4.14 Each sample batch of 20 samples or less shall include a second source Cesium – 137 laboratory control sample (LCS) that is counted in the same geometry configuration as the samples.

8.5 Continuing Calibration Check and Backgrounds

8.5.1 While the detector shall be calibrated at least monthly, it may become necessary to perform continuing calibration checks. These shall be conducted when any of the following have occurred. Any event that could effect the operation of the detector such as but not limited to; broad temperature changes, disruption of power, and/or background increases. If the checks show that the detector is not performing within 2 sigma of the know values of the calibration source, the reason shall be determined and corrective action conducted, with re-calibration to follow after mitigation.

8.5.2 Background spectrums shall be collected using the same geometry configuration as samples to be counted by collecting a spectrum that is at least as long in count duration as any sample that is to be analyzed (counted).

8.5.3 Review each background spectrum for unusual "peaking" that might indicate a contaminated chamber or detector.

8.5.4 If the background, either through control chart monitoring or observation, shows interference with sample analysis, remove detector from service and label as such until repaired or cleaned to satisfactory condition after approval from the Radiochemistry Manager.

8.6 Individual Isotopic Determinations by Calculation.

8.6.1 Individual isotopes can be determined be external calculation by establishing region of interest over known energies and determining an actual detector efficiency instead of using an efficiency curve .

8.6.2 Use the basic formula to calculate the gamma-emitting activity, A, in picocuries per volume used in "V" below as follows in picocuries per volume used in "V" below as follows:

$$A = \frac{B - C}{(2.22)\,(E)\,(V)}$$

where:

B=gross gamma count per minute.

C=count per minute of background gross gamma.

E=detecter efficiency at gamma energy.

V=sample volume.

2.22=conversion factor from dpm to pCi.

Use SOP SWL-RD-122, " Error Estimates and Minimum Detectable Activities" for those calculations using appropriate variables.

9.0 CALCULATIONS

Calculations are perform by the software.

10.0 QUALITY ASSURANCE/QUALITY CONTROL

See Standard Operating Procedure "*Quality Control for Radiochemical Analyses*", SWL-RD-113.

11.0 WASTE DISPOSAL

See Standard Operating Procedure "*Hazardous Waste Management Plan*", SWL-GA-114.

12.0 METHOD PERFORMANCE

Not applicable.

13.0 REFERENCES

13.1 SWLO Standard Operating Procedures, G-Series.

14.0 DEFINITIONS

FWHM:.....Full Width at Half Maximum

15.0 ATTACHMENTS

15.1 Attachment 1: Gamma Spec. Run Log

ATTACHMENT 1

Gamma Spec. Run Log

SOUTHWEST LABORATORY OF OKLAHOMA, INC. *and*
AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.

TM

Standard Operating Procedure

Acid Digestion for Soils and Air Filters

Document No.: SWL-RD-126

Rev No. / Date: Rev. 2.2 — 07/19/00

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APPROVALS	DATES
Procedure Prepared By	Date 7/19/00
Program Manager or Section Supervisor	Date 7/19/00
Laboratory Safety Officer	Date 7/20/2000
Laboratory QA/QC Officer	Date 7/20/00

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ACID DIGESTION FOR SOILS AND AIR FILTERS

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1.0 SCOPE AND APPLICATION

1.1 The purpose of this procedure is to provide a method for acid digestion for soil/solids and air filters.

1.2 This instruction applies to the acid digestion for soil/solids and air filters.

2.0 METHOD SUMMARY

Air filter samples and soil/solids samples are prepared as per SOP SWL-RD-115, " Solid Sample Preparation". Then a representative aliquot of the material is placed in a teflon beaker and digested in acid. The combination of acids used, breaks down the materials into free ions and liberates any organic impurities as carbon dioxide gas. The final digested material contains insolubles that are filtered out before subsequent use in analytical procedures.

3.0 HEALTH AND SAFETY

3.1 Standard radiological precautions should be observed. See Standard Operating Procedure "*Laboratory Safety Plan,*" SWL-GA-111 and "*Radiation Safety,*" SWL-RD-141.

3.2 Take all necessary precautions in regards to respirator use (see "*Respiratory Protection Plan,*" SWL-GA-130.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING & STORAGE

Not applicable.

5.0 INTERFERENCE AND POTENTIAL PROBLEMS

Not applicable.

6.0 EQUIPMENT/APPARATUS

6.1 Teflon beakers

6.2 Hotplate

6.3 Whatman 42 filter paper.

7.0 REAGENTS

7.1 De-ionized water. (DI water).

7.2 Nitric acid, 16 N HNO_3 (conc.). reagent grade

7.3 Perchloric acid, 12N $HClO_4$ (conc.). reagent grade

7.4 Hydrofluric acid, 30N HF (conc.). reagent grade

8.0 PROCEDURE

8.1 The radiochemistry analyst who is experienced in standard wet preparation methods and who is trained in the use of this procedure shall be permitted to perform this procedure.

8.2 Place entire filter or a representative soil sample of 10 grams, prepared from SOP SWL-RD-115 in a 250 ml Teflon beaker and add tracers as appropriate per individual standard operating procedures. Record this information in the preparation logbooks as prescribed by each pertinent standard operating procedure.

8.3 Cautiously add: 60 ml 16N HNO_3, 5 ml 12N $HClO_4$, 5 ml 30N HF.

8.4 Digest on hot plate until volume is reduced to 15-20 ml (3-4 hours).

8.5 Remove from hot plate, cool slightly, then add: 30 ml 16N HNO_3, 5 ml 30N HF.

8.6 Digest on hot plate until volume is reduced to 10-15 ml and HF Fumes.

8.7 Remove from hot plate, cool, dilute to approximately 50 mls with DI water with swirling.

8.8 Filter through Whatman 42 filter paper. Collect filtrate. Discard solids and filter.

8.9 Assign each filtrate a sequential identification number from the Solid Prep. Log.

8.10 Dilute filtrate up to exactly 200 mls (using a graduated cylinder) and transfer to a 250 ml plastic container for storage until needed for analysis preparation.

8.11 Label container with the following information:

Dilution ID #:
Analyst:
Date Prepared:
Sample ID Number:
Total Dilution Volume:
g/ml Equivalent: (Solids)
Volume/ml equivalent: (Filters)

8.12 Record all dilution and preparation activities in the Solids Prep. Log.

8.13 Record all results in Solids Prep. Log.

9.0 CALCULATIONS

Not applicable.

10.0 QUALITY ASSURANCE/QUALITY CONTROL

See Standard Operating Procedure "*Quality Control for Radiochemical Analyses*," SWL-RD-113.

11.0 WASTE DISPOSAL/POLLUTION PREVENTION

See Standard Operating Procedure "*Hazardous Waste Management Plan*," SWL-GA-114.

12.0 METHOD PERFORMANCE

Not applicable.

13.0 REFERENCES

- EMSL-LV-0539-17, March 1979.

14.0 DEFINITIONS

Not applicable.

15.0 ATTACHMENTS

Attachment 1: "Solids Prep. Log."

ATTACHMENT 1
Solids Prep Log

1

Solids Prep Log, SWL-RD126

SOLIDS PREP LOG.FORM (126-ATT2.DOC) SWL-RD-126, Rev.:___ Book # RD126-PRP-__1__

Verified ____

Date	Analyst	Digestion ID	Sample ID	Sample Type	Volume Digest	Final Dilution	Vol. / ml	Pre-digest tracer ID / Vol.

Southwest Laboratory of Oklahoma, Broken Arrow, OK

SOUTHWEST LABORATORY OF OKLAHOMA, INC. and
AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.

Standard Operating Procedure

Glassware Cleaning, Radiochemical Analyses

Document No.: SWL-RD-138

Rev No. / Date: Rev. No.: 1.1 — 08/22/00

OFFICIAL COPY RED STAMP INDICATES ORIGINAL

APPROVALS	DATES
Procedure Prepared By	8/22/00 Date
Program Manager or Section Supervisor	8/22/00 Date
Laboratory Safety Officer	8/28/00 Date
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GLASSWARE CLEANING: RADIOCHEMICAL ANALYSES

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1.0 SCOPE AND APPLICATION

The purpose of this procedure is to provide a step direction for the cleaning of glassware and plasticware used for radiochemical analyses.

2.0 METHOD SUMMARY

Glassware and plasticware are first rinsed after use, then hand washed with "rad" decontamination soap and soaked. After the soap soak, all items are soaked in a dilute nitric acid bath. After the nitric bath items are rinsed with tap water and then rinsed with ASTM type II water and allowed to air dry before use.

3.0 HEALTH AND SAFETY

3.1 Standard safety and radiological precautions should be observed. See Standard Operating Procedure SWL-GA-111, "*Laboratory Safety Plan*", and SWL-RD-141, "*Radiation Safety*".

3.2 Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Wearing gloves, eye protection, and a laboratory coat during extraction and analysis is advisable and should provide the proper protection.

3.3 The following chemicals have the potential to be highly toxic or hazardous, consult MSDS: Nitric Acids.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

Not applicable.

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

Not applicable.

6.0 EQUIPMENT/APPARATUS

6.1 Wash and Soak basins

6.2 Appropriate cleaning brushes, sponges, etc.

7.0 REAGENTS

7.1 ASTM type II, De-ionized Water.

7.2 Nitric Acid, reagent grade, (to produce nitric bath to less than 2 pH).

7.3 Contrad Radioactive decontamination Soap, or equivalent.

8.0 PROCEDURE

8.1 All glassware, plasticware and laboratory equipment that comes in contact with the samples that are analyzed for radiochemical analyses shall be rinsed after use into appropriate waste barrels. This first rinse is to maintain that all hazardous wastes are contained and not discharged into the sanitary sewer.

8.2 After the rinsing, each item shall be thoroughly rinsed with tap water. If the glassware is not going to be cleaned immediately, each piece shall be filled with tap water and set to the side until further cleaning is possible. Items shall not be allowed to sit "dry" as this condition can promote contaminants to become more difficult to remove later.

8.3 Items shall be thoroughly washed with Contrad soap or equivalent. The use of brushes, sponges and or other items is permitted as long as these items themselves are maintained contamination free and clean.

8.4 All washed items shall be soaked submerged in Contrad soap solution for at least 3 hours.

8.5 Remove soaked items and rinse with tap water.

8.6 Place all rinsed items into a nitric acid bath that is of pH less than 2, submerged for at least 3 hours.

8.7 After the nitric bath, remove items and rinse thoroughly with tap water to remove residual acid.

8.8 Rinse each item with ASTM type II DI water at least three times.

8.9 Place items on a clean drying surface and allow to dry.

8.10 Items shall be stored away from possible contaminants to insure that only clean items are used for laboratory radiochemical analyses.

8.11 Some glassware items may be rinsed with a very dilute HF rinse prior to use, if glassware contamination has been a problem. This rinsing shall only be performed with approval from the Radiochemistry Program Manager.

9.0 CALCULATIONS

Not applicable.

10.0 QUALITY ASSURANCE/QUALITY CONTROL

Not applicable.

11.0 WASTE DISPOSAL/POLLUTION PREVENTION

See Standard Operating Procedure "*Hazardous Waste Management Plan*", SWL-GA-114.

12.0 METHOD PERFORMANCE

Not applicable.

13.0 REFERENCES

SWLO Standard Operating Procedures. G-Series.

14.0 DEFINITIONS

Not applicable.

15.0 ATTACHMENTS

Not applicable.

Typist: LLR

SOUTHWEST LABORATORY OF OKLAHOMA, INC. and
AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.

Standard Operating Procedure

Radiation Safety

Document No.: SWL-RD-141

Rev No. / Date: Rev. 4.0 — 09/14/00

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APPROVALS	DATES
Procedure Prepared By	Date 9/14/00
Program Manager or Section Supervisor	Date 9/14/00
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RADIATION SAFETY

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1.0 SCOPE AND APPLICATION

1.1 The purpose of this procedure is to provide general guidelines for the safe handling of radioactive samples, materials and standards. Every employee working in the Radioactive Materials Control Area (RMCA) on a regular basis will be familiar with the contents of this SOP and have completed the radiation safety training course given by the Radiation Safety Officer (RSO). The intent of this standard operating procedure is to implement these instructions and assist employees in the safe use of radioactive materials and radiation sources.

1.2 This procedure is applicable to all radiochemical and mixed sample activities performed at Southwest Laboratory of Oklahoma, Broken Arrow, Oklahoma.

2.0 METHOD SUMMARY

Not applicable.

3.0 HEALTH AND SAFETY

3.1 Refer to the current version of the "Laboratory Safety Plan" SOP and SWL-RD-141, "Radiation Safety".

3.2 Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. This may be done by wearing gloves, eye protection, and laboratory coat.

3.3 The following chemicals have the potential to be highly toxic or hazardous, consult MSDS.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Not Applicable.

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

Not Applicable.

6.0 EQUIPMENT / APPARATUS

6.1 Survey meter detectors shall be maintained at log-in for sample screening.

6.2 Swipe material - paper type material of about 45mm diameter.

7.0 REAGENTS

As stated.

8.0 PROCEDURE

8.1 Responsibilities

8.1.1 The Radiation Safety Officer (RSO) shall be responsible for enforcement of this procedure and specific safety requirements included therein.

8.1.2 Laboratory Safety Officer shall be ultimately responsible for providing a safe working environment and ensuring that all laboratory personnel comply with safety policies. The RSO shall also ensure that updated copies of references in section IX, be readily available for review by personnel.

8.1.3 The Radiochemistry Program Manager shall maintain control of radioactive samples and standards. The Radiochemistry Program Manager shall also ensure that all personnel handling these samples or standards are trained in accordance with the laboratory safety SOP and policies. The Radiochemistry Program Manager shall control access to the radioactive standard cabinet with a key. The Radiochemistry Program Manager and the RSO shall also be responsible for all reporting requirements regulated by law as applicable. and shall maintain and ensure laboratory compliance with the U.S. Nuclear Regulatory Commission.

8.1.4 The RSO or designee shall be responsible for screening and storage of radioactive samples.

8.1.5 All personnel shall comply with good housekeeping and safety practices when performing radiochemical activities. Personnel shall follow methodologies outlined in all standard operating procedures.

8.2 Precautions

8.2.1 Precautions to be used are in each of the SOP's. Do not handle the radioactive material until proper indoctrination in Radiation Safety has been obtained.

8.2.2 Caution is to be used as if the area that is to be surveyed has been grossly contaminated.

8.2.3 Note all other precautions throughout this procedure.

8.3 Instructions

8.3.1 Every employee will adhere to the Southwest Laboratory of Oklahoma policy of maintaining any and all radiation exposures to "As Low As Reasonably Achievable"-ALARA by the following:

8.3.1.1 Limiting time of exposure to radioactive materials to a minimum.

8.3.1.2 Maintaining as much distance from radioactive materials as practicable.

8.3.1.3 Use of shielding from sources of radiation.

8.3.2 The "Radiological Materials Control Area (RMCA)" is designated by placards on the doors marking the boundaries of this "controlled area"(see attached map).

8.3.3 Known radioactive materials will be maintained and stored in RMCA areas only (excluding radioactive materials subject to general license provisions as provided by 10 CFR such as Ni-63 sources used in ECDs for gas chromatography, Am-241 sources in smoke detectors, etc..).

8.3.4 Good housekeeping practices and common sense help to reduce potential hazards. This procedure shall be used in conjunction with the safety SOP. Adequate labeling of work areas, samples for analysis, aliquots, and separated fractions helps to control radiation hazards and to insure personnel safety.

8.3.5 Known radioactive standards and sources will be handled only under the supervision of the RSO or Radiochemistry Manager and shall be stored under lock and key when not in use.

8.3.6 Pregnant women shall notify the RSO or Management of declared pregnancy as soon as practicable in order to comply with minimal dose and exposure requirements as defined in 10 CFR part 20, these dose to an embryo/fetus and mother dose records shall be maintained by the RSO.

8.3.7 The RSO or designee assigned to perform sample receipt, screening and login for potentially radioactive samples will follow SOP, SWL-RD-139. If a sample exceeds acceptance limits stated in the Radioactive Materials License or laboratory internal limits as established in SOP SWL-RD143, he/she shall notify the RSO and Radiochemistry Program Manager immediately. All radioactive materials shall be labeled in accordance with SOP SWL-RD-143, "Radiation Control Levels and Monitoring".

8.3.8 While all known radioactive materials will be labeled as such, use care when non-radioactive items come in contact with radioactive substances to prevent contamination. All items that are suspected to have been in contact with radioactive materials shall be scanned with the handheld halogen quenched GM probe/ratemeter before exiting the RMCA. If readings are greater than 2 times background, determine source of reading (internal or external contamination) by performing swipes if needed and submit to the radiochemist trained to count for gross alpha/beta/gamma using limits established in SOP SWL-RD-143, " Radiation Control Levels and Monitoring" to determine release to the general laboratory or to the RMCA sample storage area. Record swipe results in the "Swipe Log".

8.3.9 RMCA "authorized" personnel are those whom have successfully completed the radiation safety training coarse described in this SOP and are allowed to work independently in the RMCA. Those who are not authorized shall not enter this area unless escorted by an authorized person. While escorting an "un-authorized" person in the RMCA, the authorized employee shall be present with the un-authorized person for the duration of his/her visit. The un-authorized employee

shall never handle any radioactive materials. The authorized employee shall make sure that upon exit from the RMCA the escorted employee scan each hand and foot in accordance with this SOP and instructions posted at the exit.

8.3.10 A number of health and contamination hazards are to be considered when working with radioactive materials. Many radioactive materials are capable of contaminating laboratories, instruments, and clothing. All radioactive materials in large concentrations are potentially hazardous because of the effects of their radiation external to their containers. To control these hazards, the following rules shall be in effect at all times.

8.3.10.1 For the case of radioactive materials that are capable of being volatilized or airborne, perform all work in a closed area for containment or hood that is equipped with HEPA filtration. Perform distillations, evaporations, and other such processes in a well-ventilated hood.

8.3.10.2 Do not bring food or liquid refreshments into a laboratory.

8.3.10.3 Do not smoke when handling radioactive materials. Smoking is not allowed in the facility.

8.3.10.4 After working with radioactive materials, wash hands thoroughly before eating or handling uncontaminated materials.

8.3.10.5 Wear gloves and lab coats when handling radioactive materials. All lab coats shall remain on the rack in the RMCA when not being used. Laboratory coats shall be screened by the RSO or designee for contamination to determine if they can be sent out for cleaning or put in the laboratory radiation solid waste category. The RSO shall scan the coats to be sent out for cleaning with a halogen quenched pancake probe/ ratemeter to determine if readings greater than background are discovered. If readings are greater than background, limits set forth in SOP SWL-RD-143 shall be used to decide whether the coats can be released as "free release" to a standard cleaning company for cleaning.

8.3.10.6 Radioactive materials shall be labeled with radioactive warning labels as per SOP SWL-143, "Radiation Control Levels and Monitoring". Materials that are not labeled should always be handled with caution due to the "unknown" nature of the contents.

8.3.10.7 Spills shall be cleaned up immediately, and all cleaning items, paper towels, bags, and gloves shall be disposed in accordance with radioactive waste disposal procedures. Report all radioactive spills to the RSO. After spill cleanup the area shall be frisked for radiation with a handheld survey probe or NaI rate meter and swipes taken for gross alpha/beta to insure that the cleaning process was adequate.

8.3.10.8 After cleanup of spills a survey and swipe test will be conducted to determine the need for further cleaning. Record results in the "Swipe Log".

8.3.10.9 All items that are suspected to have come in contact with radioactive materials shall be scanned with a hand-held probe upon exit from the RMCA with the results being recorded in the "Screening Log" provided at the exit. Gross alpha/beta swipes may also be necessary and should be coordinated with the RSO. Any elevated readings should be immediately brought to the attention of the RSO.

8.3.10.10 All laboratory instrumentation (regardless of suspicion of contamination) and/or equipment shall be scanned and swiped for contamination before being removed from the RMCA with results recorded in the "Screening Log" and the "Swipe Log".

8.3.10.11 All trash that is not suspected to have come in contact with radioactive materials and is ready for standard disposal outside of the RMCA shall be scanned with a survey meter and recorded in the "Screening Log" before being removed from the RMCA.

8.3.10.12 All solid materials that have come in contact with radioactive materials and cannot be decontaminated shall be placed and stored in the "Radioactive Waste" labeled trash cans until disposal.

8.3.10.13 Prior to disposal of empty uncontaminated containers to unrestricted areas, all radioactive material labels shall be removed or defaced to clearly indicate that the container no longer contains radioactive materials as per 10 CFR 20.1904(b).

8.3.10.14 All limitations and conditions of this SOP shall be met in handling liquid or uncontained radioactive materials and special laboratory equipment is to be used when working with these materials such as using vent hoods and glove boxes.

8.3.10.15 Safety precautions shall be observed in the movement of radioactive materials between buildings, rooms, and areas within rooms. Radioactive materials shall be handled with care when being moved. Use secondary containment containers when practical. Notify people in high traffic areas that materials are being located and to be careful. Pre-plan the movement of radioactive materials to make sure that the receiving location is available and free from potential hazards.

8.3.10.16 Radioactive materials shall be labeled and posted in secure areas where radioactive materials are stored.

8.3.10.17 Appropriate records as required by 10 CFR Parts 20 and 30, shall be maintained by the Radiation Safety Officer.

8.3.10.18 Follow the requirements for and method of performing sealed leak tests shall be performed. (see 8.15.)

8.3.10.19 Follow requirements and procedures for picking up, receiving and opening packages as per section 8.16.

8.3.11 All personnel leaving the RMCA shall scan their hands and feet on the provided Ludlum Geiger hand/foot monitors at each exit as per training provided by the RSO. Scan hands and feet at least 15 seconds to determine if elevated activities are present. Scan hands/feet for longer durations if needed to isolate "hot spots" (elevated count regions). If readings are noticeably greater than background, clean areas of elevated counts and re-scan. Use the handheld halogen quenched probe (maintained at the exit) and scan these areas to confirm that cleaning was adequate. If count levels fall back to background then personnel may exit. If scans still show elevated counts above background re-clean until levels have been reduced back to background. Normal background for these hand/foot scanning devices runs approximately 100 cpm.

8.3.12 In order to monitor the "external radiation levels; and concentrations or quantities of radioactive material and the potential radiological hazards that could be present" as per 10 CFR 20.1501, swipe tests shall be performed for the facility (outside the Radiological Materials Area) each three (3) months (see attached "Swipe Location Map (Outside RMCA Area)". Swipe tests shall be performed in the Radiological Materials Area monthly. (see attached "Swipe Location Map (RMCA Area)". Action shall be taken on swipes indicating results greater than 9 pCi/swipe (100 cm2) –alpha, and/or 300 pCi/swipe (100 cm2)-beta. These alpha/beta determinations shall be analyzed and performed as per SOP SWL-RD-117, "Operation of the LB4000 Low Background Counting System."

8.3.13 Swipe tests shall be conducted daily as needed by individuals performing work on radioactive materials, or when a spill occurs, or when contamination is suspected. The RSO shall direct additional swipe testing of laboratory benches, work areas, and sample containers as needed. The RSO shall implement technique or procedural changes as needed to prevent contamination by conferring with the Radiochemistry Program Manager. If required, a corrective action report shall be initiated. Additional swipes will be taken as needed and provide random screens in addition to the standard list.

8.3.14 Every effort shall be made to identify any locations with elevated swipe readings and have the areas cleaned to acceptable free release levels as defined in SOP SWL-RD-143 "Radiation Control Levels and Monitoring" and in compliance with occupational exposure limits set forth in 10 CFR. Results of area swipe testing as described here, shall be maintained by the RSO in the "Radiation Survey Results Notebook".

8.3.15 If radioactive contamination persists at a location. the RSO shall be notified and the area shall be marked off and labeled to warn all employees.

8.3.16 Personnel Exposure / Individual Monitoring:

8.3.16.1 The following levels of exposure shall not be exceeded: 10 CFR 20.1201(a)(1)(I) specifies an annual limit of the total effective dose equivalent being equivalent to 5 rems. 10 CFR 20.1207 states that the annual occupational dose limits for minors are 10% of the annual dose limits specified for adult workers in 20.1201. 10 CFR 20.1208 states that the licensee shall ensure that the dose to an embryo/fetus during the entire pregnancy, due to occupational exposure of a declared pregnant woman, does not exceed 0.5 rem.

8.3.16.2 10 CFR 20.1502 requires that individual monitoring of external and internal occupational dose are required to demonstrate that the following groups expect not to receive greater than the following levels of exposure/ intake of radioactive materials.:

8.3.16.2.1 Exposure to Radioactive Materials (from sources external to the body)

8.3.16.2.1.1 Adults likely to receive, in 1 year a dose in excess of .5 rems.

8.3.16.2.1.2 Minors likely to receive, in 1 year a dose in excess of .05 rems.

8.3.16.2.1.3 Declared Pregnant Women likely to receive, in 1 year a dose in excess of .05 rems. (See 10 CFR 20.1208 for additional requirements.)

8.3.16.2.2 Intake of Radioactive Materials

8.3.16.2.2.1 Adults likely to receive, in 1 year, an Annual Limit of Intake (ALI) in excess of 40 pCi, and that the Derived Air Concentration (DAC) shall remain below 2.00E -4 pCi/L of air. (The 40 pCi value sighted above is 10% of the ALI, as defined in 10 CFR, table 1, Column 1, mixed unknown radionuclides, of appendix B to 20.1001-20.2401 which is 400 pCi, and the 2.00E-4 value is the DAC, as defined in 10 CFR, table 1,Column 3, mixed unknown radionuclides, of appendix B to 20.1001-20.2401).

8.3.16.2.3 In order to demonstrate compliance through monitoring that 1) Exposure to radioactive materials, and 2) Air concentrations of radioactive materials are less than the required limits set forth

in 10 CFR 20.1502 "Conditions requiring individual monitoring of external and internal occupational dose" and 10 CFR 20.1201 "Occupational dose limits for adults". SWLO shall do the following:

8.3.16.2.3.1 Maintain and monitor "area dosimetry" reports to show continuous monitoring of dose equivalent for worst case scenario approach. Area dosimetry shall be placed throughout the RMCA to continuously monitor radiation levels present.

8.3.16.2.3.2 Calculate the Derived Air Concentration (DAC) through air monitoring in work areas to provide actual measurements of radioactive contaminants that may have become airborne.

8.3.16.2.3.3 Follow the rigorous swipe testing of the facility as required by this SOP and document that loose contamination is at a minimum.

8.3.16.2.3.4 Monitor the facility radioactive materials inventory to demonstrate, if necessary, worst case scenarios based on total radioactive materials on site at any given time and to maintain that the likelyhood of exceedance of limits is at a minimum.

8.3.16.3 While SWLO expects not to exceed the limits set forth in 10 CFR 20.1501 and to require individual monitoring of external occupational dose, SWLO may elect to supply certain individuals with personal dosimetry badges as an added measurement of dose equivalent in order to maintain an overall commitment to the ALARA policy. If dosimetry is worn for any reason the following rules shall be followed:

8.3.16.3.1 The RSO shall be responsible for administrating the program in accordance with 10 CFR, including issuing and collecting personal TLD badges and maintaining exposure files.

8.3.16.3.2 TLD's shall be provided and analyzed by an independent, outside contractor certified to provide radiation exposure monitoring services as specified in 10 CFR part 20.

8.3.16.3.3 Employees shall have access to their exposure histories in accordance with 10 CFR.

8.3.16.3.4 TLD badges will be worn by each assigned employee at all times when in the facility.

8.3.16.3.5 TLD badges will be placed on the storage racks when the employee leaves the facility.

8.3.17 Radiation Safety Training.

8.3.17.1 The basic course in radiation safety procedures for SWLO employees (or any individual) routinely working in or frequenting the SWLO restricted area known as the "Radiological Materials Control Area" (RMCA), is designed to fulfill the requirements of Nuclear Regulatory Commission rules and regulations (10 CFR 19.12, " Instructions to workers") for those personnel working with or near sources of radiation. The intent of this course is to implement these instructions and assist employees in the safe use of radiation sources and radioactive materials and is to be commensurate with potential radiological health protection in the restricted area (RMCA).

8.3.17.2 This training coarse is to be given prior to occupational exposure and a "refresher course" at least annually thereafter.

8.3.17.3 This training coarse is a combination of presenting required information in printed form, oral presentation, visual demonstration, and viewing of appropriate supplemental videos with an emphasis on instructor / trainee dialogue.

8.3.17.4 Objectives of the coarse are to develop a basic understanding of:

8.3.17.4.1 Dose, radioactive materials and radiation with appreciation for the relative hazards of radiation exposure.

8.3.17.4.2 Good practices in the laboratory and accident response.

8.3.17.4.3 How to use radiation detectors and perform contamination surveys.

8.3.17.4.4 Rules, regulations, and employees rights.

8.3.17.4.5 Biological effects of exposure to ionizing radiation including special circumstances.

8.3.17.4.6 See the Appendix for actual course content and outline.

8.3.17.4.7 After successful completion of this "SWLO Radiation Safety Training Course", each participant shall be documented indicating so. (See Attachment).

8.3.18 All sealed sources containing > 10 uCi of an alpha particle emitting source, shall be leak tested at 3 month intervals. Leak testing (swiping) when needed shall be performed by the Radiochemistry Program Manager, except for the case of Ni-63 leak testing for sources used in ECD devices. Since ECD swipe testing is covered

under provisions of general NRC licensing, the manufacturers instructions and SOP SWL-RD-145, "ECD Swipe Testing for Ni-63" shall be followed.

8.3.19 Emergency Procedures for accidents involving fires, release or loss of material or accidental contamination of personnel shall be adhered to as follows:

8.3.19.1 Any small fires that involve radioactive materials in this facility shall be contained by first using non-flammable items or flame retardant materials which are supplied in the laboratory to "snuff" out the fire to prevent both the spread of the fire and possible contamination of radioactive materials. All items and areas that (after the fire has been extinguished) are possibly contaminated, shall be screened and tested as per this procedure to determine the extent and level of contamination before releasing area for normal work activities including vent stacks in the exhaust hoods. After the fire has been extinguished all personnel shall leave the immediate area until residual smoke or debris has subsided. The RSO shall be contacted immediately and shall supervise the area personally until the area has been deemed non-contaminated and ready for normal work activities. The RSO's phone number and pager number are posted throughout the RMCA. If necessary, the RSO shall flag or mark off this area to warn all employees that this area is under investigation until further action is required.

8.3.19.2 The RSO shall not allow individuals to return to areas that are suspected of containing radioactive contamination until cleaned to levels that are qualified as "free release" have been achieved.

8.3.20 The entire radiation protection program shall be reviewed for content and implementation at least annually as per 10 CFR 20.1101, "Radiation Protection Programs." Document this review by filling out Attachment 5 – "Radiation Protection Program Review Checklist."

9.0 CALCULATIONS

Not Applicable.

10.0 QUALITY ASSURANCE / QUALITY CONTROL

As per SOP SWL-RD-113, " Quality Control for Radiochemical Analyses".

11.0 WASTE DISPOSAL/POLLUTION PREVENTION

Refer to the current version of the "Hazardous Waste Management Plan" SOP SWL-GA-114.

12.0 METHOD PERFORMANCE

Not Applicable.

13.0 REFERENCES

13.1 SWLO Standard Operating Procedures, G-series, and R-series as indicated.

13.2 10 CFR, US Nuclear Regulatory Agency.

13.3 USNRC Regulatory Guide, Rev.-1, REG. GUIDE 8.29, " Instruction Concerning Risks From Occupational Radiation Exposure", February 1996.

13.4 USNRC Regulatory Guide, REG. GUIDE 8.13, " Instruction Concerning Prenatal Radiation Exposure ", December 1987 rev 2.

13.5 USNRC Regulatory Guide, REG. GUIDE 4.20, " Constraint on Releases of Airborne Radioactive Materials to the Environment for Licensees other than Power Reactors", December 1996.

13.6 Texas A&M University Radiation Safety Course Materials, 1994, given by the Office of Radiological Safety (ORS), Texas A&M University, College Station, Texas.

13.7 29 CFR

14.0 DEFINITIONS

14.1 ALARA - As Low As Reasonably Achievable - Policy of always striving to keep any and all radiation exposures to as low as possible. This policy effectively reduces risks associated with working with radioactive materials.

14.2 RSO - Radiation Safety Officer

14.3 TLD - Thermoluminescent Dosimeter

14.4 Survey - Means an evaluation of the production, use, release, disposal, and/or presence of sources of radiation under a specific set of conditions to determine actual or potential radiation hazards. When appropriate, such evaluation includes, but is not limited to, tests, physical examination of location of materials and equipment and measurements of levels of radiation or concentration of radioactive material present

14.5 Swipe Testing - Wiping of a work area with a clean swab for the purposes of obtaining a sample during a survey to determine a level of or a lack of loose radioactive contamination. A swipe is considered approximately 100 cm2. Also referred to as wipe testing.

14.6 Contamination - Radioactive material in a place where it is not wanted.

14.7 Handheld Probe Detectors/ Ratemeters - Device that employs an electric field that causes the tube to discharge when radiation ionizes a neutral gas molecule. Depending on the construction they can be used to detect alpha, beta, and/or gamma radiation. These detectors are not capable of energy discrimination.

14.8 RMCA - Radiological Materials Control Area. Controlled area for the use and handling of radioactive and potentially radioactive materials. Doorways to these areas are marked as "Radiological Materials Control Area".

15.0 ATTACHMENTS

15.1 Radiation Safety Training Course Content / Outline – Attachment 1

15.2 Facility Floor Plan – Swipe Location Map (Outside RMCA Area) – Attachment 2

15.2.1 Facility Floor Plan Key – Attachment 2A

15.3 RMCA Floor Plan – Swipe Location Map (RMCA Area) – Attachment 3

15.3.1 RMCA Floor Plan Key – Attachment 3A

15.4 SWLO Radiation Safety Training Certificate – Attachment 4

15.5 Radiation Protection Program Review Checklist – Attachment 5

Attachment 1
Radiation Safety Training Course Content / Outline

This outline is the framework from the RSO or designee shall instruct the radiation safety training course with the main topics that are to be covered. (All coarse handouts. information. and videos are maintained by the RSO and are available for review upon request):

I. Nature of Radioactivity

A. What is Radiation:

1) Atomic and Nuclear Structure

a) Atomic Structure (Provide handout with atom diagram described)

1) 92 naturally occurring elements and 11 man-made

2) Major Atomic Components - Protons. Neutrons. and Electrons

3) Atomic Configuration

4) Nuclear Structure. Nomenclature

5) Atomic Number, Atomic Mass

6) Isotopes

7) Nuclear Changes - Radioactivity

2) Characteristics of Radioactive Emissions / Definitions

a) Radioactivity - Process of nuclear decay or disintegration whereby an unstable isotope (nuclei) releases energy in the form of particles and /or electromagnetic radiation.

b) Radiation - particles and/or electromagnetic energy emitted by an atom (usually in the decay process).

1) Alpha Radiation - large particle equivalent to a helium nucleus without its electrons.

2) Beta Radiation - small particle having the mass and charge equal in magnitude to those of an electron.

3) Gamma Radiation - short wave length electromagnetic radiation (photon).

4) Other forms of radiation.

c) Radioactive Material - substance made up wholly or in part of radioactive atoms (radioisotopes or radionuclides).

d) Radioactive Decay - a spontaneous nuclear transformation characterized by the emission of energy and or mass from the nucleus and usually accompanied by a transformation from one element to another.

e) Half-life - time required for 1/2 of radioactive atoms to decay.

1) Half Life Calculations

2) Activity

f) Curie (Ci) - unit of radioactivity equal to 3.7 X 1010 disintegration's per second.

g) Electron Volt (eV) - Unit of Energy - the computed potential energy of one electron at a potential of 1 Volt.

h) Radiation Symbols - (emission Greek letter symbols. etc..)

B. Radioactive Interactions

1) Alpha Radiations -

a) Shielded by 1 sheet of paper. Will only travel short distances (in air at STP only several centimeters).

b) Alpha particle itself not able to be inhaled.

2) Beta Radiations -

a) More penetrating than alphas. Will travel over one meter in air at STP.

b) Can give off X-rays (Bremstrahlung) when inter-acting with shielding material.

3) Gamma Radiations -

a) Will travel great distances even through various materials. Shielded best with high compacted materials such as lead or concrete.

4) Tissue Interactions

a) Alpha - Not an external hazard... sensitive tissues shielded by dead layer of skin.

Significant internal hazard: Once ingested, sensitive tissues exposed because there is no protective skin tissue. Note: cannot inhale alpha particles

Typical Alpha emitters at SWLO: Uranium, Thorium, Plutonium, Radium Radon..... Almost any isotope with Z> 83.

b) Beta - Not always shielded by dead layer of skin on body, sensitive tissue (primarily skin) can be exposed. Is an external hazard. Is an internal hazard (not as great as alpha):

Typical Beta emitters at SWLO: Strontium, Tritium.

c) Gamma (photons)- Is an external hazard. Is an internal hazard (but less than alpha and beta). Shielded best by high density materials (like lead).

Typical Gamma emitters at SWLO: Cesium, Cobalt, Potassium.

II. Radiation Detection

A) Handheld Survey Instrumentation (Fixed and Loose Contamination, etc..)

1) Halogen Quenched pancake probe / Ratemeter

2) 1 x 1 NaI handheld Gamma Detector / Ratemeter

B) Stationary Hand/Foot Survey Instrumentation / Ratemeter

C) Contamination Surveys

1) Loose Contamination Swipe Surveys, 100 cm2 moderate pressure. gross alpha. beta and gamma.

D) Background Radiation

1) Parents of the four natural decay chains:

a) U-238: half-life. 4.51 E 9 years

b) Th-232: half-life. 1.41 E10 years

c) Pu-241: half-life, 13.2 years

d) U-235: half-life, 7.1 E 8 years

2) Other prominent naturally occurring radioactive materials:

a) H-3 (tritium)

b) K-40

c) C-14

3) Nuclear weapons programs. Chernobyl

4) Consumer Products

5) Medical Radiation Exposure

III. Control of Radioactive Materials

A) Radioactive Materials Control Area

B) Materials Labeling

C) Incoming Materials Screening

D) Exit Screening

E) Radioactive Materials Inventory

F) Materials Containment

G) Waste Segregation, and Waste Disposal Procedures.

H) Housecleaning, (Cleanliness will be emphasized to minimize contamination.)

I) Materials having activities greater than 1 uCi total activity shall only be handled by the Radiochemistry Program Manager, and or Radiation Safety Officer.

J) Only properly trained individuals should attempt decontamination and recovery operations. The Radiochemistry Program Manager and/or the Radiation Safety Officer shall personally monitor and supervise/instruct the decontamination and or recovery operations at this facility.

K) All known radioactive materials shall be labeled as such. If materials are unkown, the RSO shall be notified to correct and label as needed.

L) Performance of radiation survey and monitoring procedures. (see section M-2.)

M) Safety requirements for storage of radioactive materials, including labeling of containers and posting of secure areas where radioactive materials are to be stored.

N) The maintenance of appropriate records.

O) Prohibition of frequent transfer of potentially contaminated equipment between potentially contaminated areas and unrestricted areas.

P) Emergency procedures. – Containment of spills, evacuation of contaminated and potentially contaminated areas. Emergency numbers are posted throughout the RMCA which include pager numbers for the RSO, Radiochemistry Program Manager, Hazardous Materials Cooridinator, and the Laboratory Safety Officer. The RSO shall be notified as soon as possible of any unusual events involving radioactive materials.

Q) Requirements and procedures for picking up, receiving, and opening packages.

IV. Exposure Control and Monitoring

A) Exposure Monitoring (External Dose)

1) Area TLD (Thermoluminescent Dosimetry), Continuous Monitoring

2) Personal TLD if necessary.

B) Internal Monitoring

1) By Demonstration (Radionuclide Air Monitoring Program)

2) Bioassay if necessary.

V. Waste Handling

A) Radioactive Waste Segregation (solids and liquids)

B) "Liquid Zero Effluent Discharge Policy"

VI. Exposure and Dose

A) Exposure - Measure of charge produced by radiation interacting in air.

1) Unit - Roentgen (R)

2) Typically use mR (10 E -3 R)

3) Exposure Rate - mR/hr

a) 1 mR/hr is a low exposure rate

b) 100 mR/hr is a high exposure rate

B) Dose - Measure of energy deposited per unit mass of any medium (usually interested in tissue).

1) Unit - RAD (Radiation Absorbed Dose)

2) Typically use mRAD (10 E -3 RAD)

3) Dose Rate - mRAD/hr

a) 1 mRAD/hr is low dose rate

b) 100 mRAD/hr is high dose rate

4) Dose Equivalent - Measure of Biological Effect

a) Unit - REM (Roentgen Equivalent Man or Mammal)

b) Typically use mrem (10 E -3 REM)

c) Dose equivalent rate - mrem/hr

i) 1 mrem/hr is low dose equivalent rate

ii) 100 mrem/hr is high dose equivalent rate

5) For Most of Our Needs -- betas and photons (gammas)

1 R = 1 RAD = 1 REM, & 1 mR/hr = 1 mRAD/hr = 1mREM/hr

6) Neutrons and alphas particles are the main exceptions to 5 above.

C) Annual Occupational Dose Limits

1) Adults

a) 5 rem/yr to whole body or 50 rem/yr to any individual organ or tissue other than the eye.

b) 15 rem/yr to the lens of the eye.

c) 50 rem/yr to the skin or extremities.

2) Minor - 10 % of adult limits.

3) Declared pregnant female

a) Dose to embryo/fetus = 0.5 rem during entire gestation period

b) Dose should be uniformly distributed over gestation period.

4) Dose Limits for Members of the Public.

a) .1 rem/yr

b) Dose rate not to exceed 0.002 rem/hr (2 mrem/hr)

VII. Risks from Occupational Radiation Exposure

A) Review of NRC Regulatory Guide 8.29

B) Review of NRC Regulatory Guide 8.13 (Prenatal Radiation Exposure) if applicable.

VIII. Record Keeping

A) Dosimetry Records

B) Swipe and Survey Records

C) Radioactive Materials Inventory

D) Records of Compliance

E) Air Monitoring Records

IX. SWLO Policies

A) Notice to Employees (NRC form 3)

B) Southwest Laboratory of Oklahoma, Standard Operating Procedures. Read SOP SWL-RD-141, "Radiation Safety".

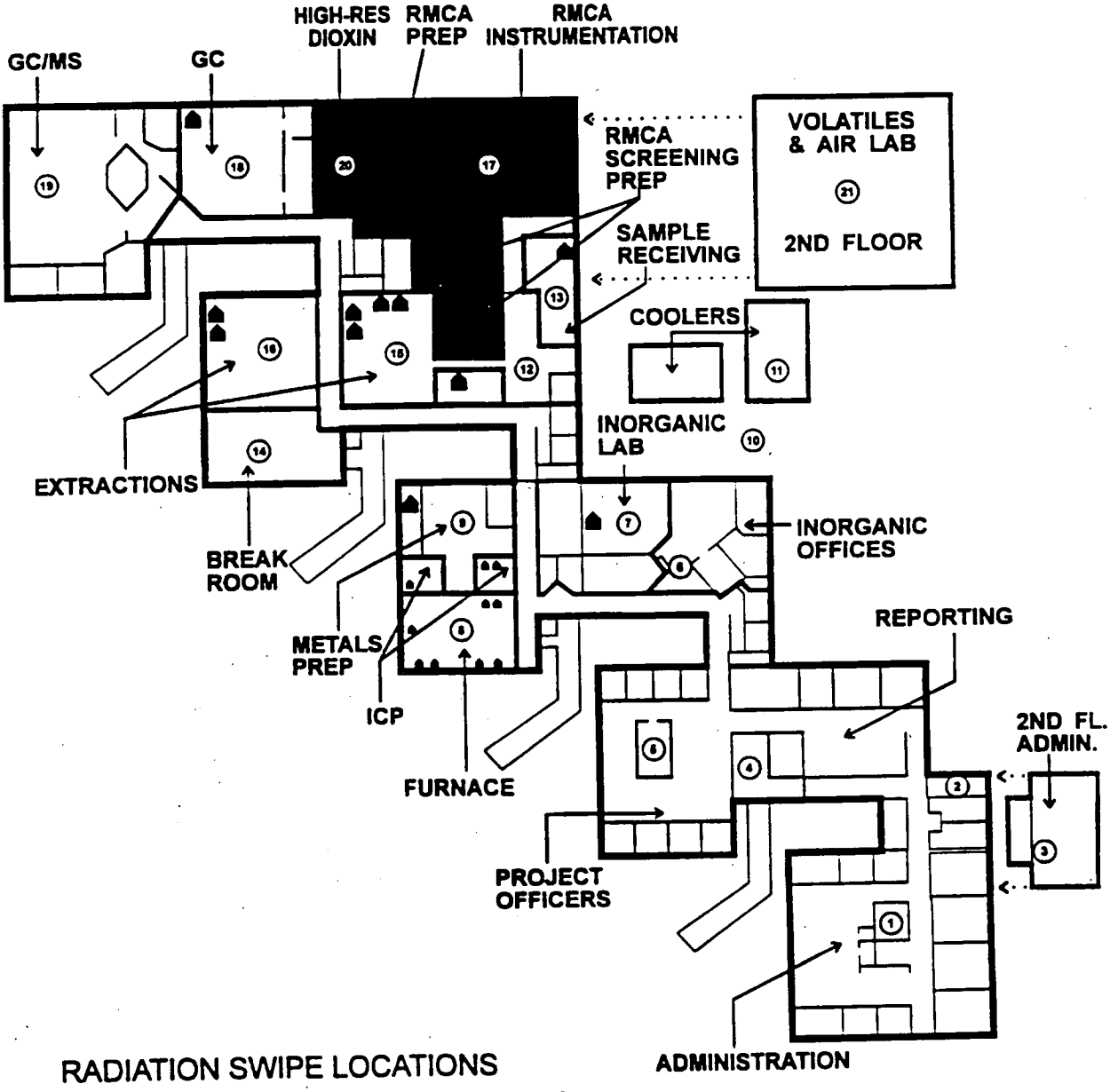
X. Film

A) "Fundamentals of Radiation Safety", Radiological Training Services. or equivalent.

XI. Discussion / Review / Test

Attachment 2
Facility Floor Plan

Swipe Location Map (Outside RMCA Area)



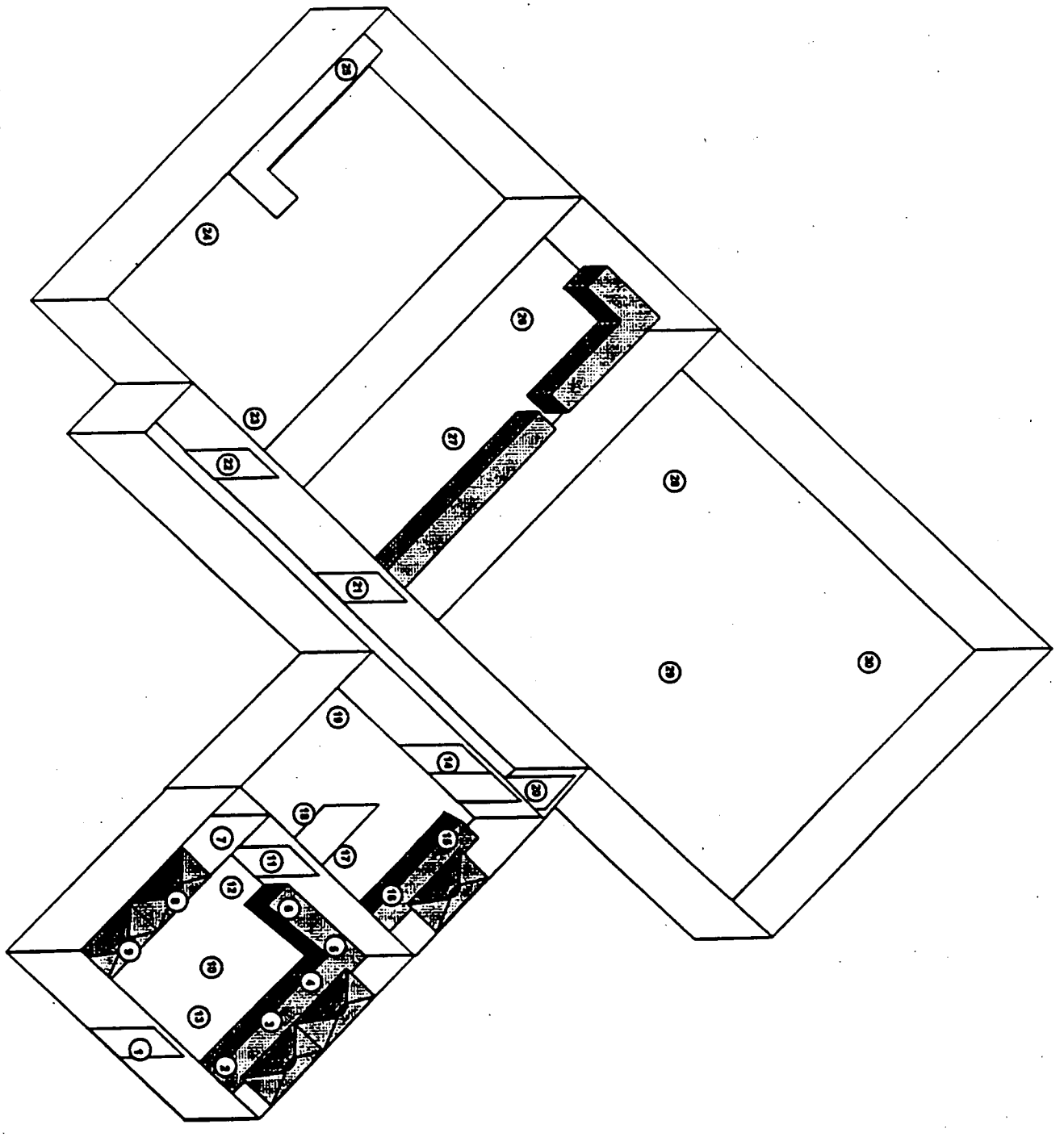
RADIATION SWIPE LOCATIONS

Layout of Southwest Laboratory's 36,000 square foot facility located in Broken Arrow, Oklahoma.

Attachment 2a
Facility Floor Plan Swipe Key

Location #	Description
Fac-1	Admin. Copy Machine and Counter Top
Fac-2	Admin. Break Room. Counter Tops
Fac-3	Upstairs Conference Room. Door Handles and Table Top
Fac-4	Reception Desk and Door Handles
Fac-5	Project Officer Copy Machine and Counters
Fac-6	Inorganic Data Area, Door Handles, desk tops
Fac-7	Inorganic Prep Lab Bench Tops and Door Handles
Fac-8	Metals Instrumentation (AA, ICP, Furnace) Bench tops / Computer Keyboards
Fac-9	Metals / Cyanide / Mercury Prep. Room, Bench Tops / Door Handles
Fac-10	Waste Storage Area, Barrel Tops, Door Handles
Fac-11	Radioactive Sample Storage, Door Handle. Rack Tops. Floor
Fac-12	Login Copy Machine. Door Handles. Solvent Storage Door Handles
Fac-13	Sample Receiving, Login Bench tops, Computer Keyboards. Door Handles. Hood
Fac-14	Break Room, Door Handles, Vending Machines, Refrig., Counter Tops
Fac-15	Solids Extraction Lab., Bench Tops, Keyboards, Door Handles. Hoods
Fac-16	Liquids Extraction Laboratory, Bench Tops, Keyboards, Door Handles, Hoods
Fac-17	VOA Laboratory, Bench Tops
Fac-18	GC Laboratory, Bench Tops, Keyboards, Door Handles
Fac-19	GC/MS Laboratory, Bench Tops, Keyboards, Door Handles

Attachment 3
RMCA Floor Plan
Swipe Location Map (RMCA)



Attachment 3a
RMCA Floor Plan Swipe Key

Location #	Description
RMCA-1	Prep Room, Door Handle
RMCA-2	Prep Room, Bench Top (Sink Area)
RMCA-3	Prep Room, Hood # 25
RMCA-4	Prep Room, Hood # 24
RMCA-5	Prep Room, Bench to (Muffle Furnace)
RMCA-6	Prep Room, Bench Top
RMCA-7	Prep Room, Sifting / Grinding Room, Bench Top
RMCA-8	Prep Room, Hood # 7
RMCA-9	Prep Room, Hood # 6
RMCA-10	Prep Room, Satellite Waste Storage Barrels.
RMCA-11	Prep Room, Floor (exit steps)
RMCA-12	Prep Room, Floor (exit area)
RMCA-13	Prep Room, Floor
RMCA-14	Screening Room, Door Handles
RMCA-15	Screening Room, Login Bench Top
RMCA-16	Screening Room, Login Bench Top
RMCA-17	Screening Room, Floor
RMCA-18	Screening Room, Floor
RMCA-19	Screening Room, TCLP Extraction Area
RMCA-20	RMCA Exit, Door Handles, GM Probes (egress frisking)
RMCA-21	RMCA Prep Lab. Door Handles
RMCA-22	Dioxin Lab, Door Handles
RMCA-23	Dioxin Lab, Floor (entrance)
RMCA-24	Dioxin Lab, Floor, Keyboards, Bench Tops
RMCA-25	Dioxin Lab, Floor, Counting Inst. Keyboards, Bench Tops
RMCA-26	RMCA Prep, Floor, Bench Top
RMCA-27	RMCA Prep
RMCA-28	RMCA Instrumentation, Keyboards, Sample Loading Racks, Floor
RMCA-29	RMCA Instrumentation, GC/MS Bench Tops, Sample Loading Racks
RMCA-30	RMCA Instrumentation, GC, GC/MS, Bench Tops, Sample Loading Racks

Attachment 4
Radiation Safety Training Certificate

Radiation Safety Training, SWL-RD141

SWLO Radiation Safety Training Certificate (141-ATT3.DOC)

This document is to certify that the individual listed below has successfully completed the Radiation Safety Training Course and curriculum as described in SWLO standard operating procedure, SOP RD-141, "Radiation Safety".

Training Item	Date Completed	Mark (X) As Completed
I. Nature of Radioactivity		
II. Radiation Detection, Demonstration		
III. Control of Radioactive Materials		
IV. Exposure Control and Monitoring		
V. Waste Handling		
VI. Exposure and Dose		
VII. Risks from Occupational Radiation Review Reg Guide 8.29 Review Reg Guide 8.13 (prenatal)		
VIII. Record Keeping		
IX. SWLO policies, NRC form 3, SWLO SOP's		
X. "Fundamentals of Radiation Safety " Film		
XI. RMCA Tour		
XII. Review and Test		

TRAINEE : _____ TRAINER: _____

Southwest Laboratory of Oklahoma, Broken Arrow, OK

Attachment 5
Radiation Protection Program Review Checklist

Radiation Safety, SWL-RD141

RADIATION PROTECTION PROGRAM REVIEW CHECKLIST (141-ATT5.DOC)

This document is to certify that the radiation protection program has been reviewed for content and implementation.

Reviewed Contents	Date Reviewed	Initials
SOP SWL-RD-141, Section 1.0 through 7.0		
SOP SWL-RD-141, Section 8.0		
SOP SWL-RD-141, Section 9.0 through 15.0		

Notes: (Make notes of changes, or updates needed to the "Standard Operating Procedure"

_____

_____

_____

_____

_____

_____

RADIATION SAFETY OFFICER: _____ - DATE: _____

Southwest Laboratory of Oklahoma, Broken Arrow, OK

APPLICATION OF GAS CHROMATOGRAPHY/MASS SPECTROSCOPY (GC/MS) FOR THE DETECTION OF MILITARY AGENTS MUSTARD AND LEWISITE, NERVE AGENT BREAKDOWN PRODUCTS DIMP AND DMMP, AND MUSTARD BREAKDOWN PRODUCTS 1,4-DITHIANE AND 1,4-THIOXANE IN WATER AND SOIL

1.0 KEYWORDS: Gas Chromatography/Mass Spectroscopy, Lewisite, 2-Mercaptoethanol, Mustard, 1,4-Dithiane, 1,4-Thioxane, DIMP, DMMP.

2.0 Written 04AUG98 (GMD)

3.0 OFFICE OF CONTACT: Monitoring Branch, Chemical Operations Division, Chemical & Biological Defense Agency, U. S. Army, Aberdeen Proving Ground, MD 21010-5423.

Point of Contact: Mr. Tom Rosso
Telephone Number: (410) 436-8428

4.0 APPLICATION: Detection of Mustard, Lewisite, 1,4-Dithiane, 1,4-Thioxane, DIMP, and DMMP in water and soil samples.

Five concentrations were used to plot the calibration curve. The method yields a linear response.

5.0 TESTED CONCENTRATION RANGE: The approximate concentrations of the standards used to plot the curve are: 0.25, 1.25, 2.50, 5.0, and 20.0 ng/ul.

Detection Limit: The instrument detection limit is 0.20 ng/ul.

Interferences: No interferences were found. It is desirable, however, to turn off the detector while the derivitizing agent is eluting from the column.

Analysis Rate: After the GC/MS is calibrated, approximately five samples per hour can be analyzed.

6.0 SCIENTIFIC BASES: The sample is extracted in Methylene chloride with 6000 parts per million (ppm) 2-Mercaptoethanol. The extract is analyzed by GC/MS. The Mass Selective Detector (MSD) forms specific ions by electron beam bombardment, which are separated and measured.

7.0 INSTRUMENTATION: Hewlett Packard (HP) GC Model 5890 with MSD 5971/2/3.
Column: Rtx-1701 or RTx-5, 0.25 mm id, 30 meters long.
Computer: HP ChemStation.

8.0 CALIBRATORS: Solutions of above mentioned concentrations of compounds in Methylene chloride with 6000 ppm 2-Mercaptoethanol.

9.0 PROCEDURE:

9.1 Extraction: Two grams of the sample is weighed into a Pyrex tube. Two milliliters of the 2-Mercaptoethanol fortified Methylene chloride is added to the tube. The Pyrex tube is vortexed and the solvent layer is removed, filtered, and placed in a 1.8 milliliter autosampler vial. The sample concentration is 1-1.

9.2 Calibration: Load the standards into the autosampler tray. Create a sequence, run the standards, and calibrate. The correlation coefficient should be >=0.99 to continue.

9.3 GC/MS Parameters: Oven temperature program 50 deg C. Initial time 1.5 min, rate 20 deg/min, ramp to 200, hold 2 min, ramp 40 deg/min to 230 deg, hold .25 min. Total run time 12.00 minutes.

1,4-Thioxane	1,4-Dithiane	Mustard	Lewisite	DIMP	DMMP	
104	120	109	151	97	94	Target Ion
74, 60, 61	92, 59, 61	111, 158, 160	107, 212, 186	123, 79, 59	79, 109, 63	Qualifier Ions

9.4 Quality Control: Check standards are run at least every ten samples. A laboratory reagent blank (LRB) is extracted with every batch. A matrix spike and matrix spike duplicate (MS and MSD) should be extracted every 20 samples. A matrix spike amount (MSA) is prepared in exactly the same way as the MS and MSD for direct comparison. (If 50 ul of the spiking solution is added to the sample, with a final volume of 1 ml, then 50 ul of the same solution is added to 1 ml of the solvent - no theoretical values are necessary. A duplicate sample is extracted every 10. Target ions can be changed if interferences warrant the change. This is permissible if the calibration is performed under the same conditions.

9.5 Calculations: Results are usually expressed in parts per billion (ppb). The standards run are ng/ul or parts per million (ppm). Since the samples are extracted at a 1-1 concentration, the calculation is as follows:

Results (ppm) X 1000 = Results (ppb)

9.6 Holding Times: Ten day holding time for samples kept at 4 degrees Celsius. Extract holding time is 40 days.

gmd

cc TER

APPLICATION OF GAS CHROMATOGRAPHY/MASS SPECTROSCOPY (GC/MSD) FOR THE DETECTION OF THIODIGLYCOL (TDG) AND ALKYL METHYLPHOSPHONIC ACIDS IN SOIL AND WATER SAMPLES

KEYWORDS: Gas Chromatography/Mass Spectroscopy, Thiodiglycol (TDG), Methylphosphonic acid (MPA), Ethyl methylphosphonic acid (EMPA), Isopropyl methylphosphonic acid (IMPA)

1.0 Current Version: 18 Dec 2000: Original: 13 June 2000 (TER)

2.0 OFFICE OF CONTACT: Monitoring Branch, Chemical Applications Division, Edgewood Chemical Biological Command (ECBC), U.S. Army, Aberdeen Proving Ground, MD 21010-5424.

Point of Contact: Mr. Thomas E. Rosso
Telephone Number: (410) 436-8428

3.0 APPLICATION: Detection and quantification of military agents breakdown products from extraction of soil and aqueous samples.

4.0 TESTED CONCENTRATION RANGE: The concentration range of the calibration curve is 0.05 nanograms (ng) to 5.00 nanograms (ng).

Interferences: No interferences were found.

5.0 SCIENTIFIC BASIS: Water soluble chemical agent breakdown products such as TDG, MPA, EMPA, and IMPA are extracted from soil and aqueous samples. The extract is dried under a purge of nitrogen to dryness and reconstituted in a 50/50 mixture of hexane/N,O-bis(trimethylsilyl)trifloroacetamide (BSTFA). The BSTFA derivitizes the analytes of interest to form trimethylsilyl derivatives. These derivatives are easily separated and detected on a GC capillary column and mass spectrometer. In order to increase selectivity and sensitivity the mass spectrometer detection utilizes Selected Ion Monitoring (SIM) specific for each analyte.

6.0 INSTRUMENTATION:
Hewlett Packard (HP) GC Model 6890 with MSD 5973 or equivalent
Computer: HP ChemStation.

7.0 CALIBRATION STANDARDS: Calibration solutions of EMPA, TDG, and IMPA can be mixed and MPA should made and analyzed separatly. The standards should be diluted using the 50/50 BSTFA/Hexane mix. Suggested calibration concentrations for all analytes are: 0.05µg/ml, 0.10µg/ml, 0.50µg/ml, 1.00µg/ml, 5.00µg/ml.

8.0 PROCEDURE:

8.1 Extraction:

1. Two grams of soil is weighed out and extracted with 2 ml of distilled/deionized water or two milliliters is removed from the aqueous sample.
2. Filter extract with 0.45 micron Supor filter into a vial. It may be necessary to centrifuge the extract up to 1 hour prior to filtration due to suspensions.
3. Evaporate the extract under a nitrogen purge to dryness. Note the volume of extract prior to evaporation.
4. Add 1 ml of the 50/50 mix of BSTFA and Hexane to the evaporated extract and mix thoroughly.

5. Heat the extract to 60°C for 30 mins.
6. Allow sample to cool and transfer into GC vial. Sample is ready for analysis.

8.2 GC/MS Parameters

A. Oven Parameters
1. Initial Temperature – 60°C
2. Initial Time – 1.0 min
3. Time/Temperature Ramp

#	RATE	FINAL TEMP	FINAL TIME
1	20°C/min	120°C	0.00
2	40°C /min	260°C	0.50 min

4. Total Run Time – 8.00 min

B. GC Column
1. HP-5MS liquid phase or equivalent (5% Phenyl methyl siloxane/ 95% dimethylpolysiloxane)
2. 30 meter length
3. 0.25 mm inner diameter
4. 1 micron film thickness

C. Injection Parameters
1. Mode: Pulsed Splitless
2. Temperature: 250 °C
3. Pressure: 10.41 psi
4. Pulse Time: 0.50 min
5. Purge Total Flow: 30 ml/min
6. Injection Volume: 2 µl

8.3 Calibration: The mass spectrometer is tuned prior to each day with Perfluorotributylamine (PFTBA). The standards are injected directly into the injection port of the GC using the auto sampler. Five concentrations are used to plot the calibration curve. The method yields a linear response for all agents. The correlation coefficient for all curves is 0.990 or greater before analyses are begun. If the calibration is <0.990, the operator shall recalibrate the instrument, if the second calibration is <0.990, the operator must conduct some manufacture recommended maintenance prior to conducted another calibration.

8.4 Quantitation of the agents is based upon the Target Ion response, the other qualifier ions are used for confirmation only. The following table lists possible target and qualifier ions for each of the agents. NOTE: Circumstances may warrant choosing one of the qualifier ions as the target ion. This is acceptable if the calibration file is adjusted accordingly. The following table lists possible target and qualifier ions for each of the target and qualifier ions for each of the agents:

Analyte	Target Ion	Qualifier Ions	Group Start Time	Retention Time
EMPA	153	169, 181, 75	0.00 min	4.50 min
IMPA	153	169, 195, 75	0.00 min	4.65 min
MPA	147	225, 227, 240	4.75 min	4.85 min
TDG	116	147, 103, 73	5.20 min	5.76 min

The mass spectrometer is not turned on until 4.00 min to allow the excess BSTFA derivatizing agent to elute.

8.5 Quality Control:

I. Check Standards – Are run at least every ten samples. Acceptable results are 70 – 130 percent. Higher recoveries than 130% may permitted at the end of the day.

II. Matrix Spike/Matrix Spike Duplicate (MS/MSD) - The MS/MSD samples are run every 20 samples to determine the daily method recoveries. If the sample batch for the day is less than 20 samples, a MS/MSD must be performed for each batch. The two samples must be within 30% of each other or the MS/MSD's must be repeated.

8.6 Analysis Rate: After the GC/MSD has been calibrated, approximately 6 samples per hour can be analyzed.

8.7 Holding Times: The maximum holding time for samples is ten days and the maximum holding time for the derivatized sample extract is forty days when each are stored at 4 degrees Celsius.

APPLICATION OF GAS CHROMATOGRAPHY/MASS SPECTROSCOPY (GC/MS) FOR THE DETECTION OF MILITARY AGENTS MUSTARD AND LEWISITE, NERVE AGENT BREAKDOWN PRODUCTS DIMP AND DMMP, AND MUSTARD BREAKDOWN PRODUCTS 1,4-DITHIANE AND 1,4-THIOXANE IN WATER AND SOIL

1.0 KEYWORDS: Gas Chromatography/Mass Spectroscopy, Lewisite, 2-Mercaptoethanol, Mustard, 1,4-Dithiane, 1,4-Thioxane, DIMP, DMMP.

2.0 Written 04AUG98 (GMD)

3.0 OFFICE OF CONTACT: Monitoring Branch, Chemical Operations Division, Chemical & Biological Defense Agency, U. S. Army, Aberdeen Proving Ground, MD 21010-5423.

Point of Contact: Mr. Tom Rosso
Telephone Number: (410) 436-8428

4.0 APPLICATION: Detection of Mustard, Lewisite, 1,4-Dithiane, 1,4-Thioxane, DIMP, and DMMP in water and soil samples.

Five concentrations were used to plot the calibration curve. The method yields a linear response.

5.0 TESTED CONCENTRATION RANGE: The approximate concentrations of the standards used to plot the curve are: 0.25, 1.25, 2.50, 5.0, and 20.0 ng/ul.

Detection Limit: The instrument detection limit is 0.20 ng/ul.

Interferences: No interferences were found. It is desirable, however, to turn off the detector while the derivitizing agent is eluting from the column.

Analysis Rate: After the GC/MS is calibrated, approximately five samples per hour can be analyzed.

6.0 SCIENTIFIC BASES: The sample is extracted in Methylene chloride with 6000 parts per million (ppm) 2-Mercaptoethanol. The extract is analyzed by GC/MS. The Mass Selective Detector (MSD) forms specific ions by electron beam bombardment, which are separated and measured.

7.0 INSTRUMENTATION: Hewlett Packard (HP) GC Model 5890 with MSD 5971/2/3.
Column: Rtx-1701 or RTx-5, 0.25 mm id, 30 meters long.
Computer: HP ChemStation.

8.0 CALIBRATORS: Solutions of above mentioned concentrations of compounds in Methylene chloride with 6000 ppm 2-Mercaptoethanol.

9.0 PROCEDURE:

9.1 Extraction: Two grams of the sample is weighed into a Pyrex tube. Two milliliters of the 2-Mercaptoethanol fortified Methylene chloride is added to the tube. The Pyrex tube is vortexed and the solvent layer is removed, filtered, and placed in a 1.8 milliliter autosampler vial. The sample concentration is 1-1.

9.2 Calibration: Load the standards into the autosampler tray. Create a sequence, run the standards, and calibrate. The correlation coefficient should be >=0.99 to continue.

9.3 GC/MS Parameters: Oven temperature program 50 deg C. Initial time 1.5 min, rate 20 deg/min, ramp to 200, hold 2 min, ramp 40 deg/min to 230 deg, hold .25 min. Total run time 12.00 minutes.

1,4-Thioxane	1,4-Dithiane	Mustard	Lewisite	DIMP	DMMP	
104	120	109	151	97	94	Target Ion
74, 60, 61	92, 59, 61	111, 158, 160	107, 212, 186	123, 79, 59	79, 109, 63	Qualifier Ions

9.4 Quality Control: Check standards are run at least every ten samples. A laboratory reagent blank (LRB) is extracted with every batch. A matrix spike and matrix spike duplicate (MS and MSD) should be extracted every 20 samples. A matrix spike amount (MSA) is prepared in exactly the same way as the MS and MSD for direct comparison. (If 50 ul of the spiking solution is added to the sample, with a final volume of 1 ml, then 50 ul of the same solution is added to 1 ml of the solvent - no theoretical values are necessary. A duplicate sample is extracted every 10. Target ions can be changed if interferences warrant the change. This is permissible if the calibration is performed under the same conditions.

9.5 Calculations: Results are usually expressed in parts per billion (ppb). The standards run are ng/ul or parts per million (ppm). Since the samples are extracted at a 1-1 concentration, the calculation is as follows:

Results (ppm) X 1000 = Results (ppb)

9.6 Holding Times: Ten day holding time for samples kept at 4 degrees Celsius. Extract holding time is 40 days.

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