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Quality Assurance Project Plan Addendum No. 1

for

Mustard Gas Burial Ground (SWMU 01)

RCRA Corrective Measures Study

Volume 2 of 2 - Appendix D

Naval Surface Warfare Center Crane

Crane, Indiana

Southern Division
Naval Facilities Engineering Command

Contract Number N62467-94-D-0888

Contract Task Order 0315

January 2004

APPENDIX D

STANDARD OPERATING PROCEDURES FOR LABORATORY
(INCLUDES ONLY SOPs THAT CHANGED SINCE APPROVED QAPP)

LABORATORY STANDARD OPERATING PROCEDURES (SOPs)
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SOP No.	REV No.	QAPP/WP	TITLE
PACE LABORATORIES			
SWL-GA-100	10.1	131[1]	Laboratory Quality Asssurance Manual
SWL-GA-101	3.1	131[1]	Preparation, Review, Revision and Control of Documents
SWL-GA-102	5.0	131[1]	Information Systems Quality Assurance Program
SWL-GA-104	8.0	131[1]	Personnel Training
SWL-GA-105	4.0	131[1]	Laboratory Corrective Action
SWL-GA-106	3.0	131[1]	Archival and Retrieval of Sample Documentation
SWL-GA-108	5.3	131[1]	Performance & System Audits
SWL-GA-109	1.3	131[1]	Traceability of Standards
SWL-GA-110	4.1	131[1]	Laboratory Documentation of Sample Custody
SWL-GA-112	5.0	131[1]	Control Charts
SWL-GA-113	5.1	131[1]	Definition and Determination of Detection Limits
SWL-GA-115	9.0	131[1]	Sample Custodian
SWL-GA-116	4.0	131[1]	Bottle Preparations
SWL-GA-120	4.3	131[1]	Calibration of Electronic Balances
SWL-GA-121	3.2	131[1]	Glassware and Labware Cleaning
SWL-GA-124	3.1	131[1]	LIMS for Sample Receiving
SWL-GA-127	4.0	131[1]	Preventive Maintenance
SWL-GA-128	5.1	131[1]	Maintenance of "Zone" Log Books
SWL-GA-133	2.1	131[1]	Usage of Blanks and Backup Blanks
SWL-GA-135	2.0	131[1]	Sample Login Review
SWL-GA-139	3.0	131[1]	Maintenance and Management of the LIMS System
SWL-GA-140	1.1	131[1]	Homogenization of Soil, Sediment, and Sludge Samples
SWL-IN-301	5.4	315[4]	Anions in Water by Ion Chromatography
SWL-IN-304	6.0	315[4]	Nitrate-Nitrite/Nitrite (Colorimetric, Automated, Cadmium Reduction)
SWL-IN-310	5.5	131[1]	Total Organic Carbon in Water
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SWL-OV-304	4	131[1]	Analysis of Volatile Organics by GC/MS (8260B-25 mL Purge)
SWL-RD-113	1.4	131[1]	Quality Control for Radiochemical Analyses
SWL-RD-124	4.1	131[1]	The Preparation and Tracking of Standards and Tracers
SWL-RD-125	1.0	315[4]	Gamma Spectrometer Operation/Calibration

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SOP No.	REV No.	QAPP/WP	TITLE
SWL-RD-138	1.1	131[1]	Glassware Cleaning, Radiochemical Analyses
SWL-RD-141	4.0	131[1]	Radiation Safety
MICROSEEPS LABORATORY			
SOP-ADM10	1	315[4]	Standard Operating Procedure for Glassware Washing
SOP-AM20GAx	2.1	315[4]	Analytical Method AM20GAx Standard Operating Procedure for the Analysis of Biodegradation Indicator Gases
SOP-PM 01G	1.1	315[4]	Standard Operating Procedure for the Preparation of Samples for the Analysis of Biodegradatoin Indicator Gases
SOP-AM21G	1.1	315[4]	Standard Operating Procedure for the Analysis of Volatile Fatty Acids in Water
SOP-SM 9	1.2	315[4]	Standard Operating Procedure for the Collection of Dissolved Gases from Water Using the "Bubble Strip" Sampling Technique at the Well Site

All laboratory SOPs associated with analysis to performed for SMUW 01 have been revised or have not been included in previous QAPPs.

1 "Quality Assurance Project Plan for Mustard Gas Burial Ground Resource Conservation and Recovery Act Phase III RCRA Facility Investigation," NSWC Crane, June 2001.

2 "Quality Assurance Project Plan Addendum No. 1 for Mustard Gas Burial Ground (SWMU 01)" NSWC Crane, October 2003.

SOP No.—standard operating procedure number.
REV No.—SOP revision number.
QAPP—quality assurance project plan.
WP—work plan.

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GA-100 Part 1 of 3

The Standard Operating Procedures (SOPs) contained herein are adopted by Pace Analytical Services.

These SOPs will be followed until superceded by Pace Analytical Services SOPs.

Signature:	Signature:	Signature:
Chuck Hoover	Roger C. Splinter	Dave Neal
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SOUTHWEST LABORATORY OF OKLAHOMA, INC. *and*
AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.

Standard Operating Procedure

Laboratory Quality Assurance Plan

Document No.: SWL-GA-100

Rev No. / Date: Rev. 10.1 — 08/11/03

1 of 3

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APPROVALS	DATES
Melissa Chance Procedure Prepared By	8-11-03 Date
Robert Mathis Laboratory Manager or Director	8/14/03 Date
Chuck Hoover Laboratory Safety Officer	8/11/03 Date
Chuck Hoover Laboratory QA/QC Officer	8/11/03 Date

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

Document Status

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1.0 QUALITY ASSURANCE

1.1 Introduction

This document details the quality assurance system employed by Southwest Laboratory of Oklahoma, Inc./American Analytical and Technical Services, Inc. (located in Broken Arrow, Oklahoma). Figure 1.1 on the following page shows our affiliate and field office locations in the laboratory network. The Broken Arrow laboratory has been carefully constructed and organized to operate as a single entity. This document details laboratory procedures used at our Broken Arrow facility. The emphasis is on the Quality Assurance requirements based on such agency guidelines as the USEPA Contract Laboratory Program, Navy Installation Restoration Laboratory Quality Assurance Guide, US Army Corps of Engineers shell for Chemical Analytical Services, the Air Force Center for Environmental Excellence Quality Assurance Plan, NELAC (National Environmental Laboratory Accreditation Conference), and DOE EMCAP (Environmental Management Consolidated Audit Program). It defines the system(s) used in identifying, documenting, and implementing project specific requirements. The purpose of this manual is to assure data of a stated quality and defensibility are being reported by SWLO/AATS.

1.2 Corporate QA Policy Statement

The management has directed all of the staff to maintain a system which ensures the generation of consistent and accurate data which meets the data quality objectives of our clients, and for the various programs in which we participate.

1.3 Quality Assurance

The Quality assurance program is part of the sound analytical protocol used by individuals and laboratories to detect and correct problems in the measurement process or to demonstrate attainment of a state of statistical control. The objective of the quality assurance program is to reduce measurement errors to agreed-upon limits and to produce results of acceptable quality. Two concepts are involved in quality assurance: (1) quality control, the mechanism established to control errors, (2) quality assessment, the system used to verify that the analysis is operating within acceptable limits..

1.4 Quality Control

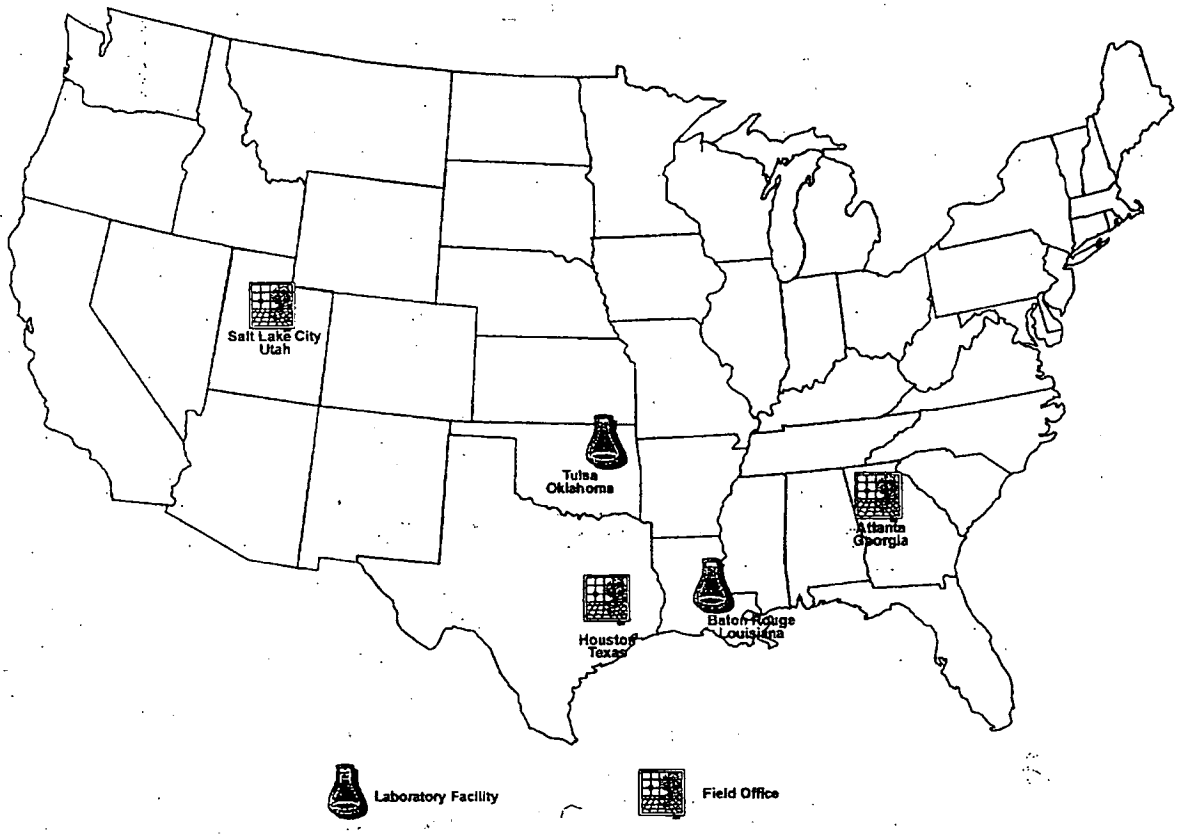
The quality control program includes the following:

- Development of and strict adherence to principles of good laboratory practice.
- Consistent use of standard operation procedures.
- Establishment of and adherence to carefully designed protocols for specific measurement programs,
- Reliable and well-maintained equipment,
- The close supervision of all operations by management and senior personnel.

When properly conceived and executed, a quality control program will result in a measurement system operating in a state of statistical control, which means errors have been reduced to acceptable levels and have been characterized statistically.

Figure 1.1

Southwest Laboratory and Affiliate Locations



1.5 Quality Assessment

Quality assessment includes a variety of techniques required to assess the quality of the measurement process and the results. The establishment of a system of "control charts" is a basic principle. Control charts are plots of multiple data points from the same or similar sample processes versus time. They are used to determine if a system is in a state of statistical control. Control charts are used to visualize or monitor the relative variability of repetitive data. They are used as appropriate with reference materials, spike samples, and the analysis of surrogates as a means to assessing the accuracy of measurements.

1.5.1 Quality Assessment Procedures

Procedures used to assess the effectiveness of the quality control system include:

1.5.1.1 Internal Performance Audits conducted by the QA Officer to assure adherence to the LQAP, and SOPs to assure systems are performing correctly. The use of control samples, replicate measurements, and use of reference materials in conjunction with control charts are also used to monitor the system performance.

1.5.1.2 External Performance Audits-conducted by the use of inter-laboratory checks such as:

1) Participation in laboratory evaluation programs; State Programs (Florida, Arizona, Oklahoma, etc.) USEPA CLP Organics and Inorganics, and US Army Corps of Engineers' HTRW Program, DOE's, QAP, and MAPEP Studies.
2) Participation in WP & WS Performance Studies.

Using the assessment process, the overall quality system is maintained. Problems identified are routed through the Corrective Action Process, through which implementation of specified corrective actions improve the overall quality system. The quality system will be monitored using internal audits and blind PE's.

1.5.2 Quality Assessment Procedure Summary

The basic quality control program and its effectiveness, shall be maintained on a daily basis by tracking the following parameters:

- Laboratory Control Samples.
- Areas of the internal standard for specific organic analysis.
- Surrogate spike recovery for organic analysis.
- Matrix spike and matrix spike duplicate sample results for accuracy and precision.
- Managerial Quality Systems Assessment is performed weekly as per Section 18.2 and SOP SWL-GA-143.

1.6 QA Review

Assembled data shall be reviewed by the Project Officer (PO) before technical compilation into contract deliverables. Final review of about 8 - 12% of the assembled deliverables package is performed by the Quality Assurance Officer.

1.7 Data Quality

Data quality is the totality of features and characteristics of data that bear on their ability to satisfy a given purpose. Measurements of major importance are accuracy, precision, completeness, representativeness, and comparability, (see Section 10.3.2. for definition).

1.8 Quality Assurance Plan Review

An overall review of the Laboratory Quality Assurance Plan (LQAP) is performed on an annual basis to ensure that the document adheres to current principles of good laboratory practice. The LQAP is revised more frequently, if necessary, and is reviewed by the Laboratory Operations Manager and the Quality Assurance Officer. Review and revision of the LQAP generally includes:

- Any procedure or policy changes
- Changes in Organizational Chart or Resumes
- Changes in responsibilities

- Any revisions to certifications and accreditations
- Changes in the facility or layout
- Changes in equipment or instrument inventory
- Changes to internal laboratory forms
- Changes to external and internal quality control checks
- Performance and system audit upgrades
- Method detection limits updates
- Typographical errors
- Grammar or context errors

Revisions are documented and placed in the LQAP file. The revised LQAP is then reviewed again to ensure that the changes are correct. The final published document is then placed under document control and released to the laboratory. Outdated copies of the LQAP are archived.

1.9 Contract Review/Project Management

1.9.1 New work is brought into the Laboratory via the Marketing Dept's responses to and awards of IFBs (Invitation for Bid), RFPs (Request for Proposal), and RFQs (Request for Quotation.)

These bid packages are logged into the Marketing Department's proposal log and assigned a consecutive proposal number. The bid packages are copied and copies are distributed to applicable personnel for comments/questions. The Marketing Dept. collects comments/questions and submits them to the client for clarification and/or as exceptions to bid items.

When contracts are awarded the Marketing Dept. reviews the contract to verify that the exceptions are addressed and alert the Project Officer of any issues involved.

The detailed procedures for contract review and project initiation/management are found in SWL-GA-144, "*Review of Proposals, Bids, and Contracts*", and in the project management SOP SWL-GA-131, "*Project Management*". A Project Officer (PO) is assigned to each project and he or she will be the client's representative for the laboratory.

1.9.2 Upon award of a project or at a client's request; where and when their requirements depart from our QA Plan and SOPs, the P.O. can initiate a "GP" series procedure. The departures are drafted by P.O., QAO and/or Program Managers into a "GP-SOP". Upon approval by the P.O., Laboratory Director/Manager, QAO, and Client, the GP-SOP is presented to the appropriate personnel for training. Once personnel are trained regarding this procedure, its' guidelines are carried out with the analyses of the client's procedures.

The case narrative regarding this work contains a reference to this procedure and provided as a deliverable with the data.

Details of the GP-Series SOPs can be found in GA-101 "*Preparation, Review, Revision, and Control of Documents.*"

1.9.3 The PO works with the client to determine the type and amount of sample containers that are needed, the parameter lists for analyses, methodology and quality control requirements. The PO will supply the sample custodian with an "Analytical Request Form" (see Fig.1.2) along with any special project requirements. The special requirements will be attached to the laboratory worksheets, which are distributed to the applicable departments. The PO is responsible for monitoring the project from the time the samples are received until the final report is submitted to the client.

1.9.4 He is also responsible for communicating the project requirements and changes to those requirements to laboratory personnel. When a project requires a departure from normal quality system procedures, the Project Officer initiates a new "GP" Series SOP. Appropriate personnel are trained on the project-specific SOP to ensure meeting the client's needs.

1.9.5 The PO is the first line of communication with the client and in so doing they must communicate with the laboratory any relevant concerns or complaints. Complaints are relayed to the QA Officer via e-mail. This/these e-mail message(s) is/are printed out and becomes part of the QA Dept.'s weekly report. The complaint is discussed during the weekly operations meeting with the Lab Director, Lab Manager, Program Managers, and Supervisors. Decisions are made regarding whether Non-Conformances have been encountered and whether a N.C. Report with re-training and/or a Corrective Action will be required. Details regarding the procedure of handling these issues are presented in SWL-GA-145, "Handling Complaints."

Figure 1.2

Analytical Request Form

SOUTHWEST LABORATORY OF OKLAHOMA

ANALYTICAL REQUEST FORM, SWL-GA-131

Ref# 5387
Pg 1

Client: ACCOUNTING OFFICER
Contact: Carolyn Mack
Project: OLM04.2
Program:
Location: 2950 Open
SDG: G04T

Client Code: EPA
Phone&Fax#: 703-284-9323 703-264-9222
PO Number:
Receipt Date: 07/18/2001 Due: 07/31/2001
Sample TAT: Fax Rpt 14

Reports:
Data Pkgs: CLP: 2
EDD:

Special Provisions: Region Case # 2950
Cooler temp.2.8
To date: 16-waters +MS/MSD

Client ID	Type	Matrix	MS320									
G04T5		W	X									
G04T6		W	X									
G04T7		W	X									
G04T8		W	X									
G04T9		W	X									
G04W0		W	X									
G04W1		W	X									
G04W2		W	X									
G04W3		W	X									
G04W4		W	X									
G04W5	N1	W	X									
G04W5MS	MS1	W	X									
G04W5MSD	SD1	W	X									
G04W6		W	X									
G04W7		W	X									
G04W8		W	X									
G04W9		W	X									
G04X0		W	X									

Reporting Address: ACCOUNTING OFFICER
Attn:;FINANCIAL MANAGEMENT DIVISION,ACCOUNTING OFFICE MD-32,RESEARCH TRIANGLE

Billing Address: Same As Reporting Address

Project Officer: HARRY M. BORG

Prepared 07/18/2001

2.0 ORGANIZATION AND RESPONSIBILITIES

2.1 Corporate Organization

SWLO/AATS is located at 1700 West Albany, Broken Arrow, Oklahoma 74012, with Affiliate Labs in Baton Rouge, Louisiana and St. Louis, Missouri.

Specific QA/QC responsibilities are summarized in the following subsections. Figure 2.1 shows the line-staff relationships within the Broken Arrow facility. Resumes of Key Personnel and experience of all technical personnel is presented in Appendix A.

2.1.1 President

The president oversees the job performance of the Laboratory Director, Marketing Director, and Financial Officers. He gives guidance and sets priorities with respect to these duties.

2.1.2 Laboratory Director

The Laboratory Director reports directly to the President and has overall responsibility for laboratory technical quality and personnel management. This overall management involves the quality assurance of the following items:

- Deliverable reports
- Subcontractor work product if required
- Task performance of key personnel

2.1.3 Operations Manager

The Operations Manager reports directly to the Laboratory Director and assists the Laboratory Director as the need arises. He is closely involved with the day-to-day activities of sample preparation/analyses and coordinates all laboratory activities necessary to fulfill contract and QC requirements.

2.1.4 Quality Assurance Officer

The Quality Assurance Officer (QAO) is responsible for monitoring the quality of laboratory work and taking appropriate actions to ensure that quality standards are being met. The QAO reports directly to the Laboratory Director in reviewing the work of teams and individuals and to report quality problems. The QAO is responsible for the following:

- Preparation of Performance Audit Samples, insertion of Performance Audit Samples into the laboratory sample stream, and review of results.
- Overseeing the laboratory's participation in external PE programs and following up with corrective actions when unacceptable scores are obtained.
- Coordinating external Quality Assessment audits and initiating corrective actions in response to deficiencies identified during the audits.
- Preparing and revising the Laboratory QA Plan (LQAP).
- Establishing QC procedures and reviewing warning and control limits for every test to assure they meet the reference method limits.

- Monitoring compliance with the LQAP by:

  1) Reviewing QC-related activities and documentation.

  2) Identifying and referring any instances in which QC objectives are not met to the section heads or the laboratory director for corrective action; Following up on these referrals to ensure that QC objectives are once again being met.

  3) Perform system and technical audits.

  4) Reviewing corrective action reports for out-of-control events and implementation of those Corrective Actions to improve/maintain the overall quality system.

  5) Review Non-Conformance Reports (NCR) and ensure training is conducted (where applicable), to avoid re-occurrence of NC.

- Maintaining documentation of method capability and proficiency.
- Communicating complaints received from the POs to Management.
- Oversight of Laboratory Training Program.
- Oversight of Laboratory Document Control Program.
- Prepare weekly QA reports to Management.

2.1.5 Technical Director

The Technical Director is responsible for research and development of new methods. The Technical Director will resolve technical issues related to GC/MS Analysis and will advise clients on technical issues. The Technical Director oversees the Dioxin and Air Toxic Programs.

2.1.6 Program Manager

The Program Manager oversees the primary functions of their department, including sample analysis, document control, data management, and client services. The Program Manager provides supervision and guidelines for sample handling and storage prior to analyses, maintenance of project files, data entries into the computer system after sample analysis, and quality review of the final data deliverables. The Program manager assures adherence to all standard operating procedures in their area. The Program manager is responsible for maintaining and compiling data for control charts, precision, accuracy and MDL data.

2.1.7 Department Supervisors/Group Leaders

The Department Supervisors and Group Leaders have primary responsibility for the technical quality of all data generated within their respective sections. In addition, they are responsible for the adherence to delivery schedules, management and utilization of manpower and the technical aspects of all Standard Operating Procedures (SOPs). They are responsible for scheduling routine

instrument preventive maintenance.

2.1.8 Analysts

The Analysts are responsible for the analysis of a sample, sample extract, or digestate on a particular piece of instrumentation. The analytical sequence is conducted according to the document SOP, which includes the required quality control monitoring. The process is documented in various run logs, including all associated Quality Control. The analysts are responsible for performing and documenting instrument preventive maintenance.

2.1.9 Sample Preparation Technicians

Sample Preparation Technicians (Organic & Inorganic) prepare samples requiring digestion, distillation, extraction, cleanup, etc. This process is documented in the various preparation logbooks, and follows method requirements. All method-specified QC is processed and documented likewise (i.e., blanks, spikes, etc.)

2.1.10 Project Officers (PO)

Project Officer — responsibilities include direct client representation within the laboratory. The PO acts in an advocacy role for the client. The PO will consult clients, as necessary, on technical aspects of the analytical results and how the results relate to the clients' needs. The PO will monitor all analytical projects as they progress through the laboratory and is responsible for communicating project requirements and changes in those requirements to laboratory staff.

2.1.11 Sample Custodian

Sample Custodian (SC) — responsibilities include receipt, inspection, and log-in of samples. The SC initiates internal chain-of-custody for samples upon log-in, monitors the sample storage areas and oversees the maintenance of internal chain-of-custody forms. The SC also moves samples in and out of archive and documents their dumping into waste drums, taken to the incinerator for disposal.

2.1.12 Data Managers

Data Managers — responsibilities include: document organization, assembly of all documents relating to contracts or projects (including project file and analytical file) to ensure clerical veracity during data handling, data assembly, and data report production and follow all associated SOPs.

2.1.13 Information Systems

Computer Services Personnel are responsible for the management and quality control of all computing systems (hardware, software, documentation and procedures), generating, updating, and performing quality control reviews of automated deliverables in accordance with SOP SWL-GA-102, "Information Systems Quality Assurance Program".

2.1.14 Information Systems Manager

The Information Systems Manager is responsible for the installation, operation, and maintenance of software and programs, generating, updating, and performing quality control reviews of analytical databases and automated deliverables in accordance with SOP SWL-GA-102, "Information Systems Quality Control Assurance Program."

2.2 Authorization of Personnel

Following is the system, in place to ensure that authorized personnel are designated to replace those personnel who are absent from the laboratory. Replacements for personnel generally flow upward on the Laboratory Organizational Chart (see Figure 2.1).

- When the Sample Custodian is absent, the Laboratory Director will designate a replacement from the trained backup Personnel.
- When an analyst is absent, the Group Leader will designate a qualified replacement or the Group Leader will replace the analyst.
- When a Group Leader is absent, the Department Supervisor will replace the Group Leader.
- When a Department Supervisor is absent, the Program Manager will replace the Department Supervisor.
- When a Project Officer is absent, the Laboratory Director will designate another Project Officer as a replacement, or the Laboratory Director will replace the Project Officer.
- When the Quality Assurance Officer is absent, the Laboratory Director will replace the Quality Assurance Officer.
- The Operations Manager will be responsible for the duties of the Laboratory Director in the Director's absence.

2.3 Stop Work Authority

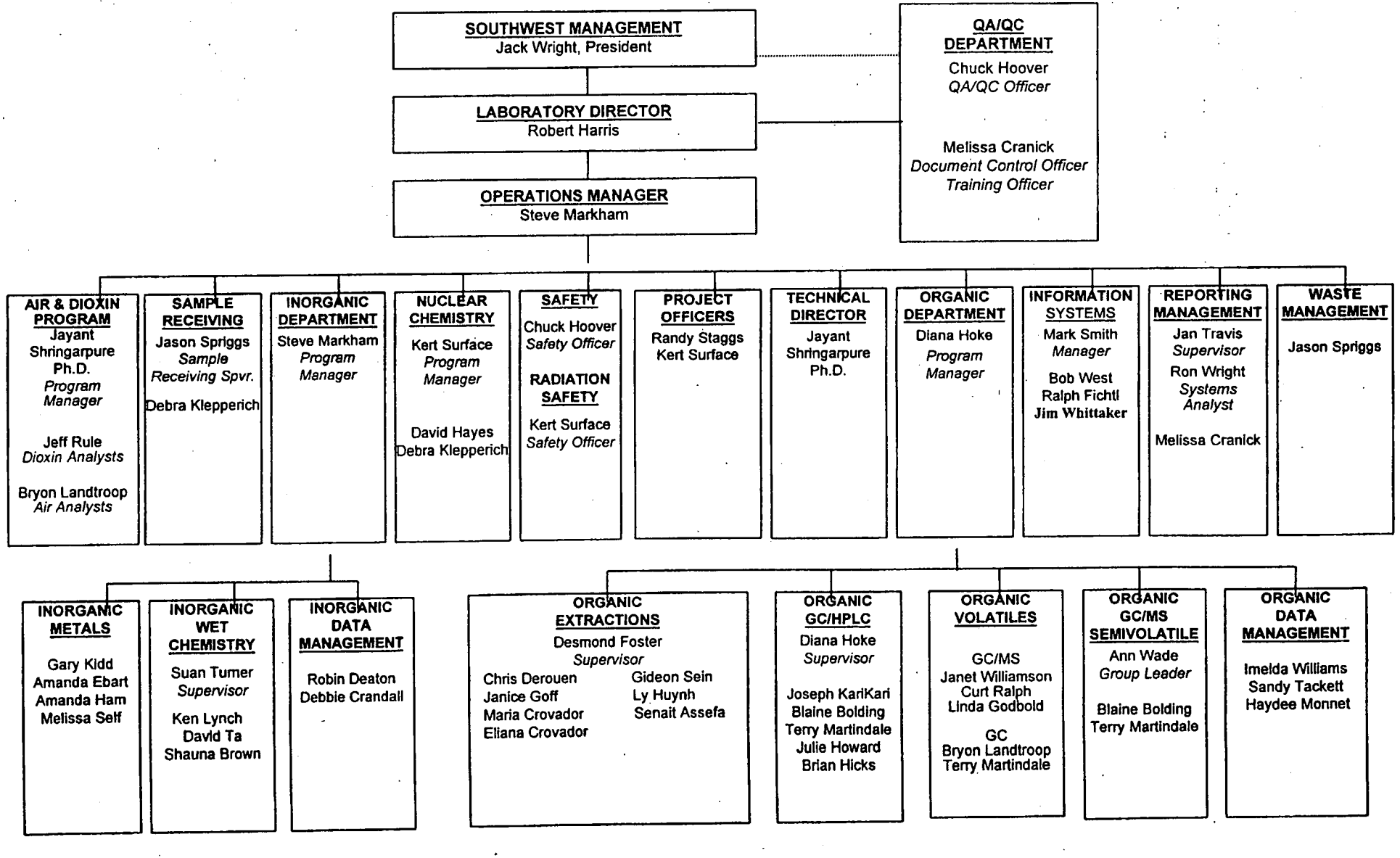
- The analyst has the authority to stop work on the task they are performing when adherence to the quality control in the SOP is not achieved. The analyst is responsible for bringing the analytical system under control and reanalyzing the affected samples. The analyst also has the authority to stop work on tasks where unsafe work conditions are encountered.
- Department Supervisors have the authority to stop work in their department when the stated LQAP or SOP policies, procedures, and practices are not met. The Department Supervisor is responsible for bringing the system back into control.
- The Program Managers have the authority to stop work for their areas when quality assurance or safety procedures are directly affected.
- The Quality Assurance Officer has the authority to stop work in all areas of the laboratory when it is deemed that a system is out of control. The QAO will meet the Laboratory Director and the Supervisor of the affected area to bring the system back under control.

- The Safety Officer has the authority to stop work when he feels the task being performed is harmful to employees.
- The President, Laboratory Director, and Operations manager have the authority to stop work for quality, safety, or financial reasons.

Figure 2.1

Organizational Chart

Southwest Laboratory of Oklahoma • Broken Arrow, Oklahoma



3.0 PERSONNEL TRAINING

3.1 Introduction

Training consists of a combination of formal sessions and "On the Job Training", both of which are detailed in SOP, SWL-GA-104, "Personnel Training". All new employees receive formal training in general orientation, safety and quality assurance as outlined in this SOP. When the training is completed, form 104-ATT1, "General, Quality Assurance and Safety Training for New Employees and/or Temporary Workers" (Fig. 3.1), is signed in acknowledgement that they received and understood the training. Those employees who will work in the radioactive materials control area will undergo further safety training in accordance with SOP, SWL-RD-141, "Radiation Safety". Supervised "On the Job Training" is provided to employees performing the technical tasks.

3.2 Safety Training

Health and safety hazards are initially introduced to all employees through a series of videotapes that cover pertinent safety topics. SOP, SWL-GA-111, "Laboratory Safety Plan" detailing the hazards involved in laboratory work, and the safeguards taken by the employees and Laboratory Management is presented to all employees at the inception of their employment. Refresher training is given to all employees on an annual basis.

3.3 Quality Assurance Training

Quality Assurance training is vital to all technical positions. The "Laboratory Quality Assurance Plan," SWL-GA-100, is discussed with all employees. The "Quality Assurance Training Checklist," shown in Figure 3.2[1] is used for this training. Annual training is conducted following the yearly update of the LQAP and prior to the LQAP effective date.

3.4 SOP Training and Proficiency Demonstration

All personnel are required to read and comply with the Laboratory Quality Assurance Plan and relevant Standard Operating Procedures. After the SOP(s) have been read and training has been completed for the appropriate analytical area, employees involved in analytical and technical data generation are certified as being proficient for those procedures. This proficiency determination is achieved by an "Initial Demonstration of Capability (IDC). IDCs will be performed by each analyst required to perform that method. Records of the IDC along with the raw data are maintained in the employees training file. Personnel shall be re-certified annually in the tasks for which they have been qualified. Details of the IDC are found in SWL-GA-104, "*Personnel Training.*"

3.5 Other Training

Records of classes taken outside of the work environment which are work related and/or aid in job performance are also maintained. These records include certificates, grade cards or transcripts from college classes or college institutions. Documents regarding job descriptions and training requirements with aspects to the employee's position are also maintained. These records are updated at least annually.

Figure 3.1

[1] A written, open-book exam is then given to the employee.

Training Documentation Forms

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. # 7.0 08/21/01

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: _____

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Figure 3.2

QA Training Checklist

QA TRAINING CHECKLIST

QA TRAINING CHECKLIST(100-ATT1.DOC) REV. #3 1–3/11/97 FORM I.D. GA-100-QATRAIN

ETHICS, POLICIES & OBJECTIVES

STANDARD OPERATING PROCEDURES (SOPs)

REVISIONS/DATES/DOCUMENT CONTROL

SAMPLE RECEIVING, SWL-GA-110

INTERNAL CHAIN-OF-CUSTODY

STORAGE OF/ PRESERVATION OF SAMPLES

LOGBOOKS

STANDARDS

ANALYTICAL

MAINTENANCE

DATA QUALITY OBJECTIVES, SWL-GA-100

METHOD DETECTION LIMITS

SOP - (SWL-GA-113)

REDBOOK

RAW DATA SUMMARY

PRACTICAL QUANTITATION LIMITS (PQLs)

BLANK CRITERIA

LABORATORY CONTROL SPIKES (LCS/LCSD) CRITERIA

MATRIX SPIKES (MS/MSD)

ANALYST REVIEW

SOP

CHECKLIST(S)

CORRECTIVE ACTIONS, SWL-GA-105

THE ABOVE ITEMS WERE DISCUSSED AND DOCUMENTS DETAILING THESE SUBJECTS WERE PRESENTD TO & REVIEWED BY THE EMPLOYEE

TRAINEE: DATE:

TRAINER: DATE:

4.0 SAMPLE CUSTODY, PRESERVATION AND TRACKING

4.1 Sample Custody

A critical aspect of sound sample collection and analysis protocols is the maintenance of strict Chain of Custody (COC) procedures. COC procedures include inventory 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]
(2) Secured to prevent tampering.
(3) Placed in a restricted area[3] by the responsible party.

The laboratory is responsible for the documentation of sample custody throughout the handling, processing, and analysis of samples.

The policies and procedures for internal and external chain of custody practices are outlined in SOP SWL-GA-110, "Laboratory Documentation of Sample Custody."

4.2 Sample Control

The Sample Control Program describes the laboratory custody procedures associated with sample receipt, storage, preparation, analysis, disposal, and security. Sample control is maintained at SWLO with several tracking systems designed to protect the integrity of the samples. Tracking systems include the use of laboratory COC procedures and sample analysis requests (in the form of worksheets), internal COC forms, the Laboratory Information Management System (LIMS), laboratory logbooks (extraction, digestion and analytical run logs). For a detailed description of the Sample Control Program see the SOPs SWL-GA-115, "Sample Custodian" and SWL-GA-124, "LIMS for Sample Receiving".

4.3 Sample Containers

During the course of a project, the client will periodically call a Project Officer with a request for sample containers. The order is taken and a "Shipping Request Form" (see Figs. 4.1 and 4.2) is filled out for either "Priority" or "Standard" shipment. Information regarding client, client contact, project reference, requested carrier, shipping address, date the containers are required, quantity requested, sampling matrix, container types and preservative requirements are recorded.

SWLO/AATS uses sample containers supplied by I-Chem and/or ESS. Certified bottles are available if requested by the client, including the necessary documentation. Bottle Certifications are reviewed by Project Officers/Purchasing Agents to verify that

---

[2] The Responsible Party is the person who has signed for custody/possession of the sample(s) or has relinquished custody back into storage areas.

[3] Restricted areas (e. g. Radiation Containment Area, VOA Refrigerator, etc.) are areas of the laboratory which have limited access exclusive to certain employees.

contamination will not effect reporting limits. All documentation of certification is maintained by the bottle shipment personnel.

The Project Officer delivers the Sample Container Request Form to the Sample Receiving department where the sample containers are prepared with the appropriate preservative, packaged (usually in coolers), and shipped with custody seals. The type of shipment, airbill, preservatives used and all other pertinent information is recorded on the request form. The request form is maintained in a file for future reference. Logs are maintained in document shipping.

4.3.1 Preservatives

Preservatives are prepared in the Inorganic or Volatile Organic department. When these preservatives are prepared, lot numbers are assigned. These reagents are checked for purity prior to use and their lot numbers are recorded on the Sample Container Request Form for tracking purposes. Table 4.1 lists the common preservatives used in bottle preparation.

Table 4.1

Preservatives

Preservative	Parameters
Hydrochloric Acid (HCl)	Volatile Organics, Total Petroleum Hydrocarbons, Oil and Grease
Nitric Acid (HNO_3)	Metals
Sulfuric Acid (H_2SO_4)	Nitrate/Nitrite, Phenols, Total Organic Chloride (TOX), Total Organic Carbon (TOC), Chemical Oxygen Demand (COD)
Sodium Hydroxide (NaOH)	Sulfide, Cyanide
Zinc Acetate	Sulfide
Sodium Thiosulfate ($Na_2S_2O_3$)	Chlorinated Drinking Water Organics

4.4 Sample Storage

All samples are stored in a secured area with separate storage facilities for volatile organics and radio chemistry samples. Unless otherwise specified by the client, all samples are stored at 2° - 6°C with a daily monitoring of all storage refrigerators. If temperatures exceed these limits, immediate corrective action is implemented to bring the system back under control.

4.5 Sample Disposal

The provisions of 40 CFR, Part 262 apply to SWLO/AATS, its procedures, policies and personnel as a generator of on-site accumulation, and transfer of hazardous waste to a transporter for off site disposal. Details of storage and disposal of samples and hazardous waste are contained in SOP SWL-GA-114, "Hazardous Waste Management Plan."

4.6 Security

Southwest Laboratory is a secured facility[4]. Samples are stored in the Sample Receiving cold storage areas except during laboratory analysis[5]. The worksheet informs the analyst which samples are needed for preparation and/or analysis. All laboratory personnel who receive samples are responsible for the care and custody of all samples. Internal Chain of Custody forms are maintained outside the sample storage area where analysts sign, initial and date the removal and return of samples from storage. Subsets (extracts and digestates) of the samples may be kept in a storage area which is controlled by the appropriate laboratory department manager. In the case of metal digestates, samples are maintained on racks between the ICP and Furnace AA analysis areas. Software security details are found in SOP SWL-GA-102, "Information Systems Quality Assurance Plan."

Figure 4.1

[4] Access is limited to employees. All doors except the front door remain locked, and the front door is monitored by the receptionist. All visitors are required to sign in and are escorted throughout their visit at the laboratory. Sample cold storage areas are secured at night and access is authorized to employees during business hours.

[5] Aqueous metals samples for Navy Projects are stored at room temperature on shelves in the Sample Receiving area unless refrigeration is specified by the project QAPP.

Shipping Containers Request Form

CLIENT SHIPPING CONTAINERS REQUEST

SAMPLE RECEIVING DEPARTMENT

Bottle Preparations SWL-GA-116, 4.0 08/02/02 File ID: GA116A1.dot White Copy — Accounting • Yellow Copy — Client • Pink Copy — File

☐ RUSH

Client Code	Project Name	Date Contacted	Req'd Ship Date	For Delivery By	Shipper Requested

CLIENT INFORMATION

Client: _____

Client Contact: _____

Phone No.: _____

Shipping Address: _____

Containers: ☐ Billable ☐ Not Billable

If NOT billable, please explain:

(RUSH Shipment Only)

Shipping: ☐ Billable ☐ Not Billable

If NOT billable, please explain:

SWL Contact:

Approved By:

SHIPPING INFORMATION

☐ Coolers (QTY. _____) ☐ Blue Ice ☐ Return Shipping Labels ☐ Make Bottle Labels

☐ Custody Seals (QTY. _____) ☐ COC's ☐ Enclose Blank Bottle Labels ☐ Attach Bottle Labels

MISC./SPECIAL PROVISIONS:

CONTAINER REQUIREMENTS

✓	QTY.	MATRIX	CONTAINERS	CONTAINER LOT #	ANALYTICAL	PRESERVATIVE	PRESERVATIVE LOT #
☐							
☐							
☐							
☐							
☐							
☐							
☐							
☐							
☐							
☐							
☐							
☐							
☐							
☐							
☐							

DATE SHIPPED	# PIECES	TOTAL WEIGHT	FILLED BY	SHIPPED VIA	SHIPPERS REF. #

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5.0 FACILITIES

5.1 Broken Arrow

SWLO/AATS's Broken Arrow facility contains 32,000 square feet of space allocated as shown in Figures 5.1 and 5.2.

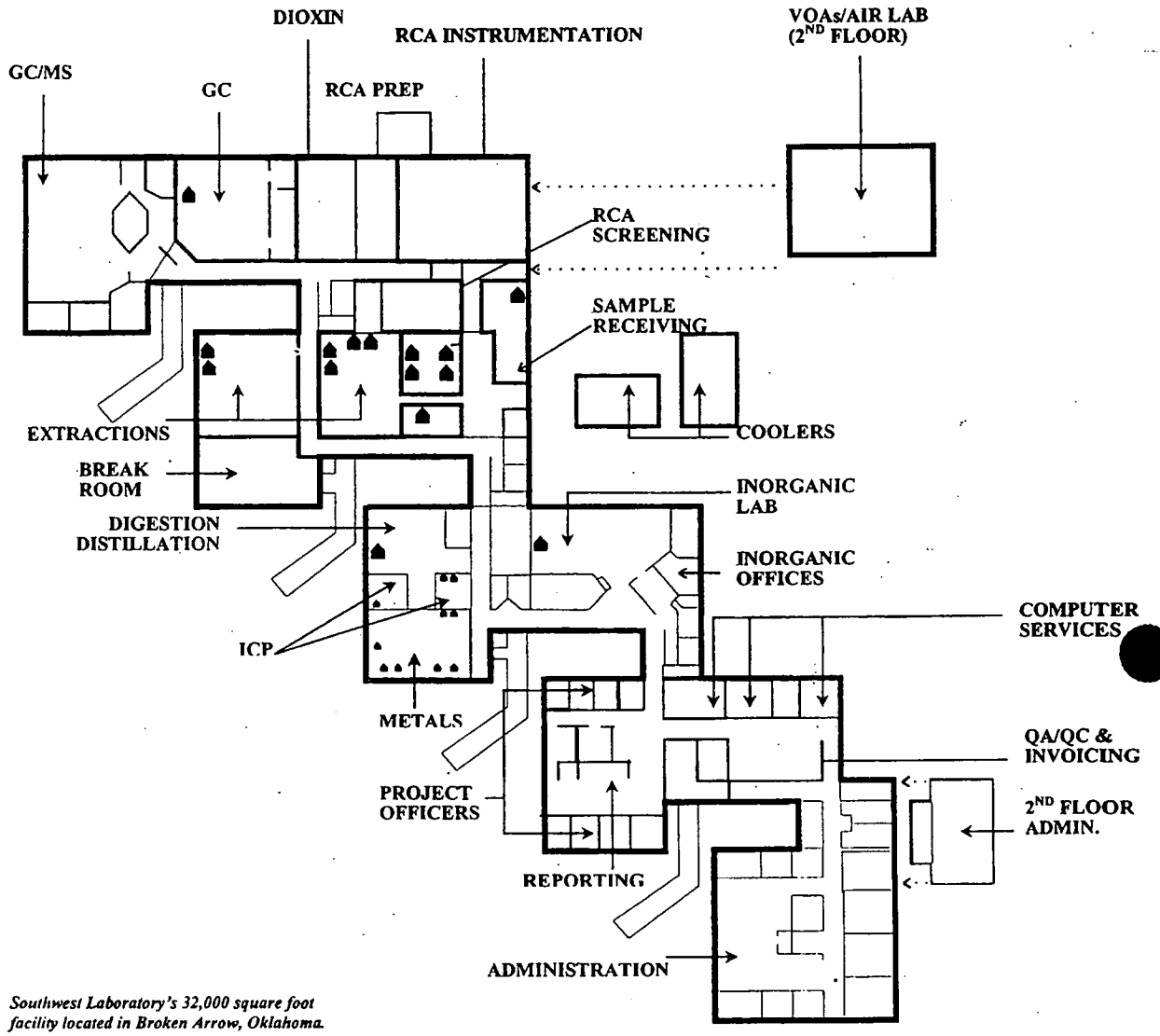
Figure 5.1

Southwest Laboratory Space Allocation

Layout of SWLO's 32,000 sq. ft. facility located in Broken Arrow, Oklahoma	Area Sq. Ft.
Volatile GC/MS	2200
Semivolatile	3200
Pesticide GC/HPLC	1100
Organic Extraction	1800
Metal Analysis	1000
Wet Chemistry	1900
Sample Receiving	1000
Radiochemistry	2600
Administrative/Reporting	4000
Dioxin	1000
Mechanical areas, walkways, and other non-work areas	14,400

Figure 5.2

Facility Layout



Southwest Laboratory's 32,000 square foot facility located in Broken Arrow, Oklahoma.

5.2 Instrumentation and Equipment

An inventory of computer hardware is found in SOP SWL-GA-102, "Information Systems Quality Assurance Plan."

Following is a current list of instrumentation and associated equipment used in providing analytical services:

Lab ID	Description	Year
70M	**GC/MS Hewlett Packard 5970 MSD** Connected to DOS Chemstation with Enviroquant data procession on HP710 Packed/Capillary Injector W/ Jet Separator Hewlett Packard 5890 Gas Chromatograph	**1986**
70F	**GC/MS Hewlett Packard 5970 MSD** Connected to DOS Chemstation with Enviroquant data procession on HP715a Packed/Capillary Injector Hewlett Packard 5890 Gas Chromatograph Hewlett Packard 7673 Auto Sampler	**1987**
70K	**GC/MS Hewlett Packard 5970 MSD** Connected to DOS Chemstation with Enviroquant data procession on HP710 Packed/Capillary Injector W/ Jet Separator Hewlett Packard 5890 Gas Chromatograph Tekmar 2000 with 2016, 2032 Auto Sampler	**1988**
70J	**GC/MS Hewlett Packard 5970 MSD** Connected to DOS Chemstation with Enviroquant Packed/Capillary Injector Hewlett Packard 5890 Gas Chromatograph Hewlett Packard 7673 Auto Sampler	**1988**
70P	**GC/MS Hewlett Packard 5970 MSD** Connected to DOS Chemstation with Enviroquant Electronic Pressure Control Capillary Injector Hewlett Packard 5890 Gas Chromatograph Hewlett Packard 7673 Auto Sampler	**1989**
70A	**GC/MS Hewlett Packard 5970 MSD** Connected to DOS Chemstation with Enviroquant Packed/Capillary Injector Hewlett Packard 5890 Gas Chromatograph Hewlett Packard 7673 Auto Sampler	**1989**

Lab ID Description Year

70C GC/MS Hewlett Packard 5970 MSD 1989
Connected to DOS Chemstation with Enviroquant ..data procession on HP715b
Packed/Capillary Injector W/ Jet Separator
Hewlett Packard 5890 Gas Chromatograph
Tekmar 3000 with 2016, 2032 Auto Sampler

70H GC/MS Hewlett Packard 5970 MSD 1990
Connected to DOS Chemstation with Enviroquant data procession on HP715c
Packed/Capillary Injector
Hewlett Packard 5890 Gas Chromatograph
Hewlett Packard 7673 Auto Sampler

71L GC/MS Hewlett Packard 5971 MSD 1990
Connected to DOS Chemstation with Enviroquant data procession on HP710
Packed/Capillary Injector W/ Jet Separator
Hewlett Packard 5890 Gas Chromatograph
Tekmar 2000 with 2016, 2032 Auto Sampler

71N GC/MS Hewlett Packard 5971 MSD 1991
Connected to DOS Chemstation with Enviroquant data procession on HP715b
Packed/Capillary Injector W/ Jet Separator
Hewlett Packard 5890 Gas Chromatograph
Tekmar 3000 with Precept II Auto Sampler

71I GC/MS Hewlett Packard 5971 MSD 1991
Connected to DOS Chemstation with Enviroquant .data procession on HP715b
Packed/Capillary Injector W/ Jet Separator
Hewlett Packard 5890 Gas Chromatograph
Tekmar 3000 with 2016, 2032 Auto Sampler

71R GC/MS Hewlett Packard 5971 MSD 1991
Connected to DOS Chemstation with Enviroquant data procession on HP715b
Packed/Capillary Injector W/ Jet Separator
Hewlett Packard 5890 Gas Chromatograph
Tekmar 3000 with Precept II Auto Sampler

70S GC/MS Hewlett Packard 5970 MSD 1992
Connected to DOS Chemstation with Enviroquant data procession on HP715a
Packed/Capillary Injector
Hewlett Packard 5890 Gas Chromatograph
Hewlett Packard 7673 Auto Sampler

Lab ID	Description	Year
72O	GC/MS Hewlett Packard 5972 MSD	1994

Connected to DOS Chemstation with Enviroquant
Packed Capillary Injector
Hewlett Packard 5890 Gas Chromatograph
Tekmar LSC 2000 with 6016 Auto Sampler

72V GC/MS Hewlett Packard 5972 MSD 1994
Connected to DOS Chemstation with Enviroquant
Packed Capillary Injector W/ Jet Separator
Hewlett Packard 5890 Gas Chromatograph
Tekmar 2000 w/2016,2032 Auto Sampler

72U GC/MS Hewlett Packard 5972 MSD 1996
Connected to DOS Chemstation with Enviroquant .data procession on HP715b
Packed/Capillary Injector
Hewlett Packard 5890 Gas Chromatograph
Tekmar LSC 3000 W/ Auto Sampler
Precept II

72T GC/MS Hewlett Packard 5972 MSD 1994
Connected to DOS Chemstation with Enviroquant .data procession on HP715c
Electronic Pressure Control Capillary Injector
Hewlett Packard 5890 Gas Chromatograph
Hewlett Packard 7673 Auto Sampler

72W GC/MS Hewlett Packard 5972 MSD 1993
Connected to DOS Chemstation with Enviroquant .data procession on HP715c
Electronic Pressure Control Capillary Injector
Hewlett Packard 5890 Gas Chromatograph
Entech 2000 Preconcentrator

72Z GC/MS Hewlett Packard 5972 MSD 1993
Connected to DOS Chemstation with Enviroquant data procession on HP715b
Packed/Capillary Injector W/ Jet Separator
Hewlett Packard 5890 Gas Chromatograph
Aerotrap Auto Sampler

70S High Resolution GC/MS VG-70S 1989
Dioxin Analysis
Connected to Vax 4000 data system
Packed/Capillary Injector
Hewlett Packard 5890 Gas Chromatograph
Fisons A200S Autosampler

Lab ID	Description	Year
Autospec	**High Resolution GC/MS Autospec**	**1989**

Dioxin Analysis
Connected to Vax 4000 data system
Packed/Capillary Injector
Hewlett Packard 5890 Gas Chromatograph
Fisons A200S Autosampler

Ultima **High Resolution GC/MS Ultima** **1998**
Dioxin/PCBs Analysis
Connected to Vax 4000 data system
Packed/Capillary Injector
Hewlett Packard 5890 Gas Chromatograph
Fisons A200S Autosampler

Ultima **High Resolution GC/MS Ultima** **1999**
Dioxin/PCBs Analysis
Connected to Vax 4000 data system
Packed/Capillary Injector
Hewlett Packard 5890 Gas Chromatograph
Fisons A200S Autosampler

Data Systems

HP Chemstation (10)
Enviroquant Software G1701AA Version 3.02

Support Equipment

Purge and Trap, Tekmar LSC2 (2)
Tekmar, ALS - 10 Position Auto Samplers (2)
Tekmar 1000 - Capillary CRYO Focuser (1)
NesLab Refrigeration Cooler (2)
Ultra Sonic Cleaner, Mettler (1)
Frigidaire Coolers - Extract Storage (2)
Tekmar LSC 2000 Purge & Trap Units (4)
Tekmar 3000 Purge & Trap Units (3)
Tekmar 2016, 16 port ALS's (5)
Tekmar 2032, 16 port ALS's (5)
Tekmar 6016 16 port ALS's
Tekmar 6032 16 port ALS's
Precept II Autosamplers (Modified) (3)
Entech 2000 preconcentrator
2016 cm Autosampler for Summa Canisters

GC Laboratory

Lab ID	Description	Year

Gas Chromatograph 1989
Carle TCD (Nat Gas)

Gas Chromatograph 1989
Carle TCD (Nat Gas)

D — Gas Chromatograph, HP 5890 1991
Tekmar 750 Headspace Autosampler

1 — Gas Chromatograph, HP 5890 ECD/ECD 1986
Dual Split/Splitless Injectors
Dual HP 7673 Auto Sampler

2 — Gas Chromatograph, HP 5890 ECD/ECD 1987
Dual Split/Splitless Injectors
Dual HP 7673 Auto Sampler

3 — Gas Chromatograph, HP 5890 ECD/ECD 1987
Dual Split/Splitless Injectors
Dual HP 7673 Auto Sampler

4 — Gas Chromatograph, HP 5890 ECD/ECD 1987
Dual Split/Splitless Injectors
Dual HP 7673 Auto Sampler

X — Gas Chromatograph, HP 5890 PID/HALL 1987
Tekmar LSC2000 Purge and Trap
Tekmar ALS 2016 & 2032 Position Auto Sampler

11 — Gas Chromatograph, HP 5890 FID 1988
Dual Split/Splitless Injectors
HP 7673 Auto Sampler

5 — Gas Chromatograph, NPD 1988
Dual Split/Splitless Injectors
HP 7673 Auto Sampler

15 — Gas Chromatograph, HP 5890 ECD/ECD 1988
Dual Split/Splitless Injectors
HP 7673 Auto Sampler

16 — Gas Chromatograph, HP 5890 Series II ECD/ECD 1989
Dual Split/Splitless Injectors
Dual HP 7673 Auto Sampler

Lab ID Description	Year
17 — Gas Chromatograph, HP 5890 Series II FID	1989
Dual Split/Splitless Injector	

Dual HP 7673 Auto Sampler

Q — Gas Chromatograph, HP 5890 PID/FID **1989**
Tekmar ALS 2016 & ALS 2032 Position Autosampler
Tekmar LSC2000

G — Gas Chromatograph, HP 5890 PID/FID **1989**
Tekmar ALS 2016 & 2032 Position Autosampler
Tekmar LSC2000
Archon 2

Y — Gas Chromatograph, HP 5890 HALL/PID **1989**
Tekmar ALS 2016 & 2032 Position Autosampler
Tekmar ALS 2000 Purge and Trap

18 — Gas Chromatograph, HP 5890 FID **1989**
Split/Splitless Injectors
HP 7673 Auto Sampler

7 — Gas Chromatograph HP-5890B ECD **1991**
Dual Split/Splitless Injectors
Twin 7673A Auto Samplers

Whatman Hydrogen Generator **1999**

Gas Chromatograph HP 5890 Series II **1998**
Tekmar 7000 Automated Headspace Analyzer

Liquid Chromatography

Lab ID	Description	Year
LC5	**Hewlett Packard HPLC (1)**	**1992**
	Variable Wavelength Detector 1050C	
	1050 Autosampler	
	HP 1050 Series Pumps (2)	
	HP 1050 solvent cabinet and column heater	
LC4	**Hewlett Packard HPLC System**	**1997**
	HP 1100 autosampler	
	HP 1100 pump	
	HP 1100 variable wavelength UV detector	
	Timberline column heater	

Lab ID	Description	Year
LC2	**Hewlett Packard HPLC**	**1998**
	Variable Wavelength Detector 1050A	
	1050 Autosampler	

HP 1050 Series Pumps (2)
HP 1050 solvent cabinet and column heater

LC3 Waters HPLC System **1999**
Waters 712 WISP (autosampler)
Waters 510 pump (two)
Waters 680 gradient controller
Waters 2487 Dual Wavelength UV detector
Waters 474 Scanning variable wavelength Fluorescence detector

Support Equipment

Oven, VWR 1310 (1)
Frigidaire Cooler - Sample Storage (1)

Extractions Laboratory

Concentrators
Kudera Danish (124) 1987
Drying Oven, Blue M 1980
Millipore (24) 1987

Extraction Apparatus
Continuous Liquid Extractors (96) 1987
Soxhlet Extractors (20) 1987
Gel Permeation Clean-up (GPC) Apparatus, ABC1002B 1998
Applied BioSystems UV Detector, Model 757 1988
Gel Permeation Clean-up (GPC) Apparatus,
ABC1002B 1991
Sontek UV Detector 1991
Zymark Benchmate 1992
SSI Pumps (2)
Sontek UV Detectors (2)
Zymark Turbovap (2) 1992
Marathon GPC 1994
Sontek Pump (1)
Sontek UV Apparatus (1)
Delfield Storage Refrigerator, Extracts
General Electric Storage Refrigerator, Standards
8-Place Auto Seperatory-funnel Shaker 1988
Glass Columns
Extraction Apparatus (20) Seperatory Funnels,
2000 ml
Funnels (75)
Dioxin Columns (80 sets)
400 ml Beakers (75)
250 ml Erlenmeyers (50)
500 ml Boiling Flask (90)
Sample Concentrator, Nitrogen Blow Down, 30 Place 1984

Sample Concentrator, Nitrogen Blow Down, 30 Place 1988
Sonic Disruptor, Sonicator (Sonics) 1989
Sonic Disruptor, Sonicator (Sonics) 1990
Sonic Disruptor, Sonicator (Sonics) 1990
Top Loading Balance, Ohaus 400 1992
Top Loading Balance, Ohaus GT 4000
Knauer UV Photometer (4)

Metal Analyses Laboratory

Atomic Absorption

Description	Year
Perkin Elmer 5100 Zeeman	1990
PE HGA 600	
PE AS-60	
Varian 400Z GFAA (2)	1992
Buck Scientific Mercury Analyzer 400	1990
Leeman PS200 Mercury Analyzer	1992
Leeman PS200 Mercury Analyzer	1993

Inductively Coupled Plasma

Lab ID	Description	Year
3	ICP 61 E Trace Analyzer Plasma Spectrometer (Thermo Jarrel Ash)	1994
	Auto sampler-TJA	
1	ICP 61E Trace Analyzer (Thermo Jarrel Ash)	1993
2	ICP, 61E Trace Analyzer (Thermo Jarrel Ash)	1994

Wet Chemistry - Inorganic

Air Compressor
Analytical Balance, Mettler H80
Balance, top-loading, Ohaus model 400 (3)
Centrifuge, Damon IEC Clinical
Conductivity Meter, HACH
Electrode (Ammonia) 95-12 Orion
Electrode (Fluoride) 94-09-00 Orion
Flow Injection Analyzer, Lachet Quikchem

Description	Year
Gooch Crucibles	
Glass Dessicators	
Hoods, Labconco - 6 Foot	
Hot Plates & Stirrers	
Ion Chromatograph, Dionex,2010i	

Dionex-DX100 Ion Chromatograph
Mettler Ultrasonic Cleaner
MuffleFurnace, Themdyne, Type 1300
pH Meter, Orion (Portable)
Plastic Dessicators, Large
Spectrometer, Bausch & Lomb, Spectronic 21
Total Organic Carbon Analyzer, Schmadzu 5050
TOX Analyzer Mitsubishi 10E-C
TOX Analyzer — Dohrmann
Carbon Analyzer Leco CR12
Dohrman Microcoulometer MCTS-20
Vacuum-Pressure Pump, Thomas
Balance-Analytical American Scientific Products
Model SP180
Balance- Top Loading; Ohaus AS120
Balance- Top Loading; Ohaus GT2100
BOD Incubator - White Westinghouse, Model:MS-U14F3BW4
BOD Incubator - E Series
PH Meter, Fisher Scientific
Centrifuge, Fisher Model: 225
IEC Clinical COD Reactor, Model: 2090-OIC
Queue Radial Shaker
Turbidmeter, HF Instruments: DRT100B
Drykeeper Desicators (10)
Blue M Oven, Stabletherm (2)
Labline, L-C- Model 2310, Model 3512
Imperial IV Water Bath
Griddles (Teflon coated hotplates for use in metals digestion) (8)

High Hazards Laboratory

Description	Year
Analytical Balance, ASP Model 2410	
Blender, Waring	
Chemical Carcinogen Glove Box, LABCONCO	
Heated Dessicator, Precision Scientific	
Shaker, Eberbach	
Special Glassware	
Columbia Fisheries Dioxin Protocol	
Sonic Disruptor, Sonicator - Heat Systems	
Muffle Furnace, Linberg	
Samsung Storage Refrigerator	
Laboratory Furniture	
1 - Four Foot Hood, KEEWANEE	
1 - Six Foot Hood, KEEWANEE	

Data Management

LIMS System

Description	Year
Computer, Gateway 2000 P5-90	1994

32 MB Memory, 1 G Hard Drive (2)
2 G Tape Backup
450 Watt Ups
Software:
- SCO Unix — Version 3.2v4.2
- TCP/IP 1.1.3F
- Informix:
  - Online — 5.00.UD5
  - 1-SQL — 4.10.UC2
  - 1-STAR — 5.00.UD5

WYSE 60 Terminals (1)

GC/MS Lab (Voa Lab Server) This system is used for retrieval of historical data only now.

Description	Year
Computer, Gateway 2000 P5-100	1996

32 MB Memory
2 G Hard Drive
2 G Tape Backup
Ups
Software:
- SCO Unix — 3.2v2.0
- TCP/IP 1.1.3F
- Informix:
  - Online — 5.00.UD5
  - 1-SQL — 4.10.UC2
  - 1-STAR — 5.00.UD5

Equipment Hardware

SWLO has four NT servers as part of its LIMS system. These systems are described below.

SWLO1-2001

AMD1.3GHz Processor

256MB RAM
(4) 60GB hard drives in RAID 0+1 configuration
Exabyte Mammoth 2 Tape Drive

Usage: Main file server, primary domain controller, backup server

SWLO2-1999
Dell 1300
(2) PIII 750 MHz Processor
512 MB RAM
(4) 9GB hard drives in RAID 5 configuration

Usage: External IIS (Web server), Exchange server, backup domain controller, proxy server, VPN server

SWLO3-1999
Dell 4500
(2)PIII 450 MHz Processor
512 MB RAM
(5) 18GB hard drives in RAID 5 configuration

Usage: File server, DHCP server, WINS server, Print Server

SWLO4-2002
AMD 900MHz Processor
512 MB RAM
(4) 30GB hard drives in RAID 0+1 configuration

Usage: Interal IIS (Web server), SQL server, Program server

Metals Lab

Description	Year
Computer, Gateway 2000 P5-90	1994

82 MB Memory
4 G Hard Drive
2 G Tape Backup
Ups
Software:
- SCO Unix 3.2v4.2
- TCP/IP 1.2.10
- Informix:
  - Online — 5.01.UD2
  - I-SQL — 4.10.UC2
  - I-STAR — 5.01.UD2

Spare

Computer, Gateway 2000 P5-60 1994
16 MB Memory
4 G Hard Drive
2 G Tape Backup
Ups
Software:
- SCO Unix 3.2v4.2
- TCP/IP 1.2.1i
- Informix:
  - Online — 5.00.UD5
  - I-SQL — 4.10.UC2
  - I-STAR — 5.00.UD5

Dev. Sys.

Computer, Gateway 2000 486DX2/50 1992
16 MB Memory
500 Hard Drive
2 G Tape Backup
Ups
Software:
- SCO Unix 3.2v2.05
- TCP/IP 1.1.3F
- CGI 1.1.02
- Informix:
  - Online — 5.00.UD5
  - I-SQL — 4.10.UC2
  - I-STAR — 5.00.UD5

Miscellaneous Computer Equipment

Terminal Ports (196)
Computer Ports (96)
Packard Bell 2400 baud modems (4)
One Hundred Computers, IBM PCs and Compatibles 1991-1996
40 MB to 2.5 GB Disk Drives
Streamer Tape Back Up System (2)

Mixed Waste Laboratory/Radiochemistry

Description	Year
Oxford HpGe Gamma Spectroscopy System 40 Percent efficient detector, Be thin window, using Oxford GammaTrac analysis software.	1995
Oxford HpGe Gamma Spectroscopy System 40 Percent efficient detector, Be thin window, using Oxford GammaTrac analysis software.	1995
Oxford Oasis Alpha Spectroscopy System 20 Chamber/Detector series using TC256 NIM modules.	1995
Oxford LB4100 Low Background Gas Proportional Alpha/Beta Counting System Multi-setector system running 8 detectors in two drawer assemblies.	1995
Packard Tri-Carb 2550 TR/LL Liquid Scintillation Counting System	1995
The Nucleus Sodium Iodide Gamma Detector System 4 Nal Detector array system using DMR-II Digital Multiplexer Router and associated analytical software.	1995
Ludlum Radon Flask Counter, Lucas Cell Counter (7)	1995
ChemCheck Kinetic Phosphorescence Analyzer ChemCheck KPA-11.	1995
Ordela Photon-Electron-Rejecting Alpha Liquid Scintillation (PERALS) Spectrometer	1995
Ludlum Handheld 1X 1 Nal microR Survey Meter	1995
Ludlum Model 3 GM Survey Meter with Pancake Probe (2)	1995
Ludlum Model 177 Scaler with Hand Frisk Platform (2) Platform – 2 each, pancake type halogen quenched G – M.	1995
Ludlum Model 177 Scaler with Hand Frisk Platform (2) Platform – 3 each, pancake type halogen quenched G – M.	1995

Support Equipment

Description	Year
Accumet pH Meter (2)	
Denver Instruments Analytical Balances (2)	
Mettler PM2000 Top Loader Balance	
Marathon 10K Large 32 location Centrifuge	
Fisher Centrifuges (2)	
Large Muffler Furnace	
Large Drying Oven	
Ball Mill (60 can capacity)	
Large Industrial Jaw Crusher	
Explosion Proof Freezer	
Electroplating Unit	
Tritium Distillation Column Array (8)	

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GA-100 Part 2 of 3

The Standard Operating Procedures (SOPs) contained herein are adopted by Pace Analytical Services.

These SOPs will be followed until superceded by Pace Analytical Services SOPs.

Signature:	Signature:	Signature:
Chuck Hoover	Roger C. Splinter	Dave Neal
Quality Assurance Officer	Dir. of Quality, Safety, Training	General Manager
Pace Analytical Services, Inc.	Pace Analytical Services, Inc.	Pace Analytical Services, Inc.
Broken Arrow, Oklahoma	Minneaplolis, MN 55414	Lenexa, KS

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

Standard Operating Procedure

Laboratory Quality Assurance Plan

Document No.: SWL-GA-100

Rev No. / Date: Rev. 10.1 — 08/11/03

2 of 3

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APPROVALS	DATES
Melissa Adamek Procedure Prepared By	8-11-03 Date
Robert Mathis Laboratory Manager or Director	8/11/03 Date
Chuck Hoover Laboratory Safety Officer	8/11/03 Date
Chuck Hoover Laboratory QA/QC Officer	8/11/03 Date

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

Document Status

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6.0 EQUIPMENT MAINTENANCE

6.1 Preventive Maintenance

A preventive maintenance program for the instrumentation ensures fewer interruptions of analyses, personnel efficiency, and lower repair costs. It eliminates premature replacement of parts, and reduces discrepancy among test results. It increases reliability of results. Detailed information on preventive maintenance is found in SOP SWL-GA-127, "Preventive Maintenance".

6.1.1 The laboratory has established the following preventive maintenance program:

6.1.1.1 Each type of equipment/instrument has a written Standard Operating Procedure (SOP) describing 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.

6.1.1.2 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.

6.1.1.3 Written equipment records are kept to document all inspection, maintenance, trouble-shooting, calibration, or modifications. Whenever maintenance is performed on an instrument, it is documented in a preventive maintenance logbook, which is kept near the equipment. The records contain the date (month, day, year), description of the maintenance done, the actual findings, the name of the person doing the maintenance, a statement of whether the maintenance operations were routine, and if those operations followed the written SOP and the documented return to control after maintenance was completed.

6.1.1.4 Performance criteria is established for judging when data from instrument performance checks indicate the need to make adjustments in the instrument operating conditions.

6.1.1.5 Backup instrumentation is prevalent throughout the laboratory. This ensures the client that should an instrument develop problems, the samples can still be analyzed on the backup instruments.

6.2 Chromatographic Instruments

Preventive maintenance is done through daily performance checks. Parameters such as retention time and response factors are observed and back-checked with prior operational performance. Details are found in SOPs, SWL-OP-201, "Preventive Maintenance of Hewlett Packard and Fisons Gas Chromatographs", and SWL-GA-103, "Tekmar Maintenance".

In addition, the following are done:

- Septa are replaced as needed.
- Incoming gas drying cartridges are changed whenever the color of the absorbent is noticed.
- Effluent absorbent traps are changed every month.
- Columns (GC and HPLC) are checked by performance and operating conditions when in use, or prior to use.
- Oven performance checked daily prior to use.

6.3 Gas Chromatography/Mass Spectroscopy (GC/MS)

The preventive maintenance is provided by Hewlett-Packard on a semi-annual basis.

6.4 Inorganic Instrumentation – the preventive maintenance schedules are in SOP, SWL-IN-103, "Inorganic Preventive Maintenance Schedule".

6.5 General Laboratory Equipment

Analytical balances of various capacities and operational modes are calibrated every six months, by a licensed specialist and officially recorded as verification of performance.

Balances calibrations are checked with standard Class S weights before usage.

7.0 LABORATORY METHODS

7.1 References

The laboratory follows analytical procedures based on EPA-approved methods for both inorganic and organic analyses of multimedia environmental samples. Methods used for inorganic and organic analysis of routine samples are based on guidance documents such as "SW846 Test Methods for the Evaluation of Solid Waste" (3rd Edition), 40 CFR part 136 as published in the Federal Register, "Standard Methods for the Examination of Water and Wastewater", 18th Edition, 1992", and "The Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air". Unless otherwise notified by the client, the above methods shall be followed. All methods are available to the analysts. The laboratory's Standard Operating Procedures for each method are available to clients. Table 7.1 lists the analytical protocols utilized at Southwest Laboratory. Table 7.2 lists the most common analytical methods used. Table 7.3 gives estimated monthly production capacity for the laboratories. These production numbers can vary due to matrix or method.

If typical methods are rendered ineffective by matrix interferences, or if analytical parameters (detection limits, precision, specificity, etc) would require method variance (e. g. modification of the method), the Project Officer will notify the client/contractor of the method modification. A copy of the variance will be sent to the client/contractor to seek approval of the method change. The modification request must show that the conditions for the laboratory variance are similar to the expected conditions (e. g. sampling and handling techniques, environmental matrix concentration range, interferences) in the EPA approved methods.

Changes in operations prior to instrumental analysis (e. g. sample preparation and storage) must be documented and authorized by the client.

When newly released versions of a method are promulgated, the laboratory will evaluate the significance of any modifications made to the method. The laboratory will start the process of method development , validation and proficiency testing after evaluating the demand for the method by laboratory clientele.

7.2 Analytical Protocols

An initial demonstration of method performance is made prior to using any method. This same practice applies if at any time there is a change of personnel or instrument type. This demonstration is performed by completing the following:

- Analysis of four replicate aliquots of a quality control check standard where the comparability to the method, or laboratory generated acceptance criteria, are compared for precision and accuracy in required methods.
- Analysis of a single blind performance sample which meets method acceptance criteria.
- Analysis to determine method detection limits, for applicable methods.

The Quality Assurance Officer (QAO) has overall responsibility for overseeing that the method demonstrations are performed. The Department Supervisors are responsible for the maintenance of the data for their departments and ensuring the demonstration is performed when necessary.

TABLE 7.1

Analytical Protocols Used

Standard Methods for the Examination of Water and Wastewater: APHA, AWWA and WPCG, 18th Edition, 1985.

Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, USEPA.

40CFR, Part 136, Federal Register, Volume 49, Number 209, Friday, October 26, 1984.

Methods for Chemical Analysis of Water and Wastes, USEPA, EPA-600/4-79-020, March 1979 and as amended December 1982 (EPA-600/482-055).

Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, USEPA, EPA-600/4-85-057, July 1982 and as subsequently amended.

Pesticide Analytical Manual, Volume I, Food and Drug Administration, Revised September 1977.

Recommended Methods of Analysis for the Organic Components Required for AB1803, State of California, 5th Edition, April 1986.

NIOSH Manual of Analytical Methods, US Department of Health and Human Services, 3rd Edition, February, 1984.

500 Series Methods-Drinking Water Test Methods, USEPA Compendium of Methods.

Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, USEPA, EPA-600/4-84-041, April 1984.

Annual Book of ASTM Standards, Water and Environmental Technology, ASTM, 1987, Volume 11.01 and 11.02.

Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, USEPA SW846, 3rd Edition, September 1986, and Update III, December 1996.

Statement of Work (SOW) Multimedia, Multiconcentration Organics, US EPA Contract Laboratory Program, 3/90.

Statement of Work (SOW) Multimedia, Multiconcentration Organics, US EPA Contract Laboratory Program, OLM03.2.

Statement of Work (SOW) Multimedia, Multiconcentration Organics, US EPA Contract Laboratory OLM04.2.

State of Work (SOW) Organic Analysis, Low Concentration Water OLC01.2 (10/92) and OLC02.0 (3/95).

Statement of Work (SOW) Multimedia, Multiconcentration Inorganics, US EPA Contract Laboratory Program, ILM03.1.

Statement of Work (SOW) Multimedia, Multiconcentration Inorganics, US EPA Contract Laboratory Program, ILM04.0.

Superfund Analytical Methods for Low Concentration Water for Inorganic Analysis (10/91).

Prescribed procedures for Measurement of Radioactivity in Drinking Water, EPA 600/4-80-032, August 1980

TABLE 7.2

Analytical Methods Typically used by Southwest Laboratory

INORGANICS S = Soil, Sediment, Sludge W = Water, Ground Water, Surface Water P = Petroleum

Method	Matrix	Description
EPA 305.1	W	Acidity
EPA 310.1/SM2320B	W	Alkalinity
EPA 350.2/350.3	W	Ammonia
EPA 405.1	W	BOD
EPA 300.0/9056	W	Bromide
EPA 130.2	W	Hardness
EPA 351.3	W	Total Kjeldahl Nitrogen
EPA 9071B/1664A	W/S/P	Oil & Grease
EPA 415.1/9060	W/S/P	Total Organic Carbon
EPA 365.1	W	Phosphorus, Total
EPA 420.2/9066/420.4	W/S/P	Phenols
EPA 410.1/410.2/410.4	W	Chemical Oxygen Demand
EPA 160.1	W	Total Dissolved Solids
EPA 160.2	W	Total Suspended Solids
EPA 160.3	W	Total Solids
EPA 160.4	W	Total Volatile Solids
EPA 160.5	W	Total Settleable Solids
EPA 300.0/9056	W/S	Chloride, Sulfate, Nitrate, o-Phosphate, Nitrite, Fluoride
EPA 340.2/300.0/9056	W	Fluoride
EPA 353.2/300.0/9056	W	Nitrate
EPA 354.1/300.0/9056	W	Nitrite
EPA 9010A/9012A/335.1/335.3/335.4/4500 CN E/4500 CN G	W/S	Cyanide
SM4500 B	W	Cyanide, Amenable
EPA 375.4/9038/9056/300.0	W	Sulfate
EPA 376.1/9030B	W/S/P	Sulfide
EPA 9034	W/S/P	Sulfide, Acid Soluble and Insoluble
EPA 377.1	W	Sulfite
EPA 325.3/9252A/300.0/9056/4500Cl F	W	Chloride
EPA 120.1/9050A	W	Specific Conductance
EPA 150.1/9040/9045A	W/S	pH
EPA 6010B/200.7	W/S/P	Metals by ICP
EPA 206.2/7060, 239.2/7421, 204.2/7041, 213.2/7131A 270.2/7740, 279.2/7841/200.9/218.2/7191/272.2/7761/200.9	W/S/P	Furnace AA
EPA 7196/7195/218.4/218.6	W/S/P	HexCr
EPA 245.1/245.2/7470A/7471A	W/S	Mercury by Cold Vapor
CLP ILM04	W/S	CLP Inorganics
EPA 1664	W	TRPH
EPA 1010	S/P	Ignitibility
EPA 1110	S/P/W	Corrosivity
EPA 330.4/4500 Cl	W	Chlorine, Total Residual
EPA 365.1/300.0/9056	W	Orthophosphate
EPA Reactivity, Section 7.3 SW-846	W/S/P	Reactivity
EPA 180.1	W	Turbidity
EPA 9095A	W/S/P	Paint Filter Liquids Test

SAMPLE PREPARATION

S = Soil, Sediment, Sludge W = Water, Ground Water, Surface Water P = Petroleum

Prep#/Method#	Matrix	Description
3020A/7060A, 7421, 7440, 7841	W	Acid Digestion
3050A/6010B, 7060A, 7421, 7740, 7841	S	Acid Digestion
3510C/8080A, 8081A, 8082, 8041, 8141A, 8310	W	Separatory Funnel
3520C/8080A, 8081A, 8082, 8270C	W	Continuous Liq/Liq
3540C	S	Soxhlet
3550B/8080A, 8270C, 8140, 8141, 8310, 8330	S	Ultrasonic
3580A/8080A, 8081A, 8082, 8270A	P	Waste Dilution
5030B/8010A, 8020, 8021, 8240A, 8260B	W/S	Purge & Trap
5035/8021, 8260B	S	Purge & Trap
3660B/8081A	W/S	Sulfur Cleanup
3665B/8082	W/S	Sulfuric Acid Cleanup
3640A/8270C, 8081A	S	Gel Permeation Chrom.
8330/8330	W/S	Methanol Extraction Acetonitrile Extraction
1311, 6010B, 8081A, 8260B, 8270C, 8151A, 7470A	W/S/P	TCLP
1312	W/S/P	SPLP

ORGANICS

S = Soil, Sediment, Sludge W = Water, Ground Water, Surface Water P = Petroleum

Method	Matrix	Description
EPA 8010A/601	W	Halogenated Volatile Organics by GC
EPA 8010A	S	Halogenated Volatile Organics by GC
EPA 8011	W/S	Ethylene Dibromide GC
EPA 8015B/FL PRO/CA/IOWA OA-1/OA-2/OK/TN/TNRCC/8015AZ	W/S	Total Petroleum Hydrocarbons DRO/GRO
EPA 8020/602	W	Aromatic Volatile Organics by GC/PID
EPA 8020	S	Aromatic Volatile Organics by GC/PID
EPA 8021	W/S	Volatiles by GC/PID/HALL in series
EPA 8041	W/S	Phenol
EPA 8080A/8081A/8082/608	W/S/P	Organochlorine Pesticides/PCBs
CLP OLMO1.8/OLM03.2/OLM04.2	W/S	Organochlorine Pesticides/PCBs
CLP OLCO1	W	Low Conc. Organics (Pesticides/PCBs)
EPA 8140/8141A	W/S	Organophosphorus Pesticides
EPA 8150B/8151A	W/S	Chlorinated Herbicides
EPA 8240B/8260B/624/524.2	W/S/P	Volatile Organics by GC/MS
CLP OLMO1.8/OLM03.2/OLM04.2	W/S	CLP Volatile Organics
CLP OLCO1	W	Low Conc. Organics (Volatiles)
EPA 8270B/8270C/625	W/S/P	Semivolatile Organics by GC/MS
CLP OLMO1.8/OLM03.2/OLM04.2	W/S	Semivolatile Organics by GC/MS
CLP OLCO1	W	Low Conc. Organics (Semivolatile)
EPA 8310/610	W/S	Polynuclear Aromatic Hydrocarbons by HPLC
EPA 8330	W/S	Explosives
EPA 8280	W/S	Polychlorinated Dibenzo-p-Dioxins/Furans
EPA 8290/1613	W/S	Polychlorinated Dibenzo-p-dioxins/Furans by HRMS
TO-1	AIR	Volatile organics using Tenax Adsorption
TO-3	AIR	Volatile organics using cryogenic preconcentration
TO-14	AIR	Volatile organics using summa canisters
TO-4	AIR	Pesticides/PCB's
TO-13	AIR	HPLC or GC/MS semi-volatiles
TO-9	AIR	Dioxins

RADIOLOGICAL

S = Soil, Sediment, Sludge W = Water, Ground Water, Surface Water P = Petroleum

Method	Matrix	Description
EPA 900.0 Mod./9310	W/S	Gross Alpha
EPA 900.0 Mod./9310	W/S	Gross Beta
EPA 900.1 Mod./9315	W/S	Total Radium (Alpha)
EPA 903.1 Mod.	W/S	Radium 226 (RN/DEEM)
EPA 901.1 Mod.	W/S	Radium 226 (G.Spec.)
EPA 904.0 Mod./9320	W/S	Radium 228
EPA 901.1 Mod.	W/S	Gross Gamma
EPA 906.0 Mod.	W/S	Tritium
EPA 905.0	W/S	Radioactive Strontium
EPA 905.0	W/S	Strontium 89
EPA 905.0	W/S	Strontium 90
EPA 907/908 M	W/S	Isotopic Uranium
EPA 907/908 M	W/S	Uranium 233/234
EPA 907/908 M	W/S	Uranium 235
EPA 907/908 M	W/S	Uranium 238
EPA 907/908 M	W/S	Uranium (Total)
EPA 907/908 M	W/S	Isotopic Thorium
EPA 907/908 M	W/S	Thorium 228
EPA 907/908 M	W/S	Thorium 230
EPA 907/908 M	W/S	Thorium 232
EPA 907/908 M	W/S	Thorium 234

TABLE 7.3

Estimated Monthly Production Capacity for SWLO/AATS – Broken Arrow

Department	Nominal	Peak
Organics Department		
GC/MS		
Volatiles	800	1200
Semivolatiles	500	1000
Dioxins	400	500
Air	400	600
GC/LC		
Volatiles	400	600
Herbicides	250	320
Pesticides/PCBs	450	600
Explosives	300	500
Miscellaneous	250	500
Inorganics Department		
Metals		
AA Furnace	200	250
Cold Vapor	800	1000
ICP	700	1000
Wet Chemistry		
Anions	500	750
Carbon, Organic	500	750
Cyanide	500	750
Oil & Grease	500	750
Total Petroleum Hydrocarbons	500	750
Phenols	500	650
Sulfides	500	650
Hazardous Waste Department		
GC/MS		
TCLP Extraction/Analysis	520	650
TCLP Zero Headspace	468	624

Capacity: Monthly maximum capacity

Metals: Production figures are set for samples, not individual elements. Most samples are analyzed for multiple elements.

Categories of Analysis: Production categories include various analyses of a similar nature (e. g. GC/LC Volatiles include SW846-8020, SW846-8010, EPA 601, BTEX, THM)

8.0 CALIBRATION

Calibration is the process for determining the correctness relative to physical or chemical standards used or assigned values in scales of measuring instruments. It establishes a reproducible reference point to which all sample measurements are correlated. Instruments are calibrated as per the SOP. All calibrations used are documented in run logs and calibration summary forms for both Initial and Continuing Calibrations. Section 10.0 describes the QC checks used to verify instrument calibrations.

8.1 Geiger Counter

Geiger counters are sent annually to an independent contractor for calibration.

8.2 Atomic Absorption Spectrophotometer Systems (AAS)/Inductively Coupled Argon-Plasma Emission Spectrophotometer (ICAP).

For graphite furnace AA and ICP, the instruments are calibrated daily, or each time the instrument is set up for measuring different analytes. Operating Parameters are found in the Method SOPs.

Calibration standards are prepared fresh before each analysis and are discarded after use. These calibration standards are purchased from commercial vendors and are certified by the vendor as to traceability to standard reference materials.

8.2.1 Detailed calibration procedures for metals analysis are found in the following SOPs:

- SWL-IN-202, "Mercury ILM04.1 Digestion and Analysis"
- SWL-IN-200, "CLP ICP/ILM04.1 Digestion and Analysis"
- SWL-IN-207, "Mercury SW846 Methods 7470A/7471A Digestion and Analysis"
- SWL-IN-205, "ICP SW3005A/3050B Digestion and SW6010B Analysis"
- SWL-IN-210, "GFAA SW846 7000 Series Analysis"

8.3 Spectrophotometers

The manufacturer instructions for instrument operation are followed for proper operating procedures.

Spectrophotometers are calibrated daily prior to any sample analysis. The calibration standards are prepared from reference materials or commercial standards (traceable to EPA or NIST reference materials) at a minimum of three concentrations, including a calibration blank to cover the anticipated range of measurements. The requirement for an acceptable initial calibration is a correlation coefficient equal to or greater than 0.996 (based on statistical historical data). Before sample analysis, an initial calibration verification standard is analyzed. The response of this standard must be within ±10% of the true values. If not, the instrument must be re-calibrated.

The instruments are also checked for wavelength calibration. For UV/IR instruments, polystyrene film is used to calibrate the wavelength.

All absorption cells (i.e., cuvettes, quartz cells) are kept clean, free of scratches and fingerprints, and are rinsed with the solution to be analyzed prior to use. Matched cells are checked to see that they are equivalent by placing portions of the same solution in both cells and taking several readings of the transmittance or absorbance.

8.4 Total Organic Carbon Analysis (TOC)

For TOC calibration, a known concentration of potassium hydrogen phthalates (KHP) solution is analyzed as the calibration solution on the carbon analyzer. A minimum of three calibration solutions encompassing the linear range of the carbon analyzer is prepared and analyzed daily or prior to the sample analysis. Linear regression analysis of standard concentration in ug C versus response in millivolts (mv) is done to obtain a calibration curve. The correlation coefficient must be equal or greater than 0.995 or the standards are rerun and a new correlation coefficient is calculated. Details of TOC calibration are found in the SOPs SWL-IN-310, "Total Organic Carbon (TOC) in Water", and SWL-IN-311, "Total Organic Carbon (TOC) in Soil/Sediment".

8.5 Laboratory Instrumentation — Organic Processing Section

Gel Permeation Chromatograph (GPC)

8.5.1 The following general procedure is employed for preparing the GPC system for calibration:

- *GPC calibration solutions* — Prepare a calibration solution in methylene chloride containing the following analytes at the minimum concentrations, as listed below:

Compound	Concentration mg/mL
Corn Oil	25.0
Bis(2-Ethylhexyl Phthalate)	1.0
Methoxychlor	0.2
Perylene	0.02
Sulfur	0.08

- The GPC column is calibrated daily using the following procedure:

  1) *Calibration of the column* — Load sample loop #1 with calibration solution. Switch the value so that the GPC flow is through the UV flow-through cell. Adjust the detector to produce a UV trace that meets the following requirements.

     A. BNA's — Examine the chromatogram from the Calibration Solution run. Adjust the detector and/or recorder sensitivity to meet the following criteria:

— Peaks must be observed for all components and should be symmetrical. (Figure 1)

— Corn oil and phthalate peaks must exhibit >85% resolution.

— Phthalate and methoxychlor peaks must exhibit >85% resolution.

— Methoxychlor and perylene peaks must exhibit >85% resolution.

The "dump" time is then calculated as the time in minutes from the point of injection to just after the elution of the corn oil and prior to the elution of bis(2-ethylhexyl)phthalate.

The "collect time is then initiated and continues until shortly after the elution of perylene. COLLECTION SHOULD BE STOPPED BEFORE SULFUR ELUTES.

Use a wash time of 10 minutes after the elution of sulfur.

Verify the flow-rate by collecting column eluate for 10 minutes in a graduated cylinder. The measured volume should be 45-55mL. Once the flow-rate is within this range, record the column pressure (should be 6-10psi) and the room temperature.

B. Pesticides — The procedure for the pesticides is identical with that for BNA's with the following exceptions

— Choose a "dump" time that removes >85% of the phthalate.

— Choose a "collect" time so that >95% of the methoxychlor is collected, and continue to collect until just prior to the elution time of sulfur.

8.5.2 Gas Chromatographs (GC) — The external calibration procedures are found in the following technical SOPs:

SWL-OP-403 "Analysis of Pesticides/PCBs by GC/EC by EPA OLM03.2"

SWL-OP-405 "Determination of Low Concentration Water for Pesticides/PCBs OLC02.0"

SWL-OP-407 "Analysis of Chlorinated Herbicides by GC, SW846, Method 8151A"

SWL-OP-409 "Analysis of Phenols SW846 Method 8041"

SWL-OP-410 "Analysis of Organophosphorus Pesticides by GC SW846 8141A"

SWL-OP-411 "Extraction and Analysis of Pesticides by EPA Method 608".

SWL-OP-414 "Pesticides by GC Capillary Column Technique, SW846-8081A"

SWL-OP-415 "Analysis of PCBs by GC, SW846, Method 8082"

SWL-OP-416 "Analysis of TEPHs by GC, SW846, Modified Method 8015B"

8.5.3 High Performance Liquid Chromatographs (HPLC) — The calibration procedure are found in the following technical SOPs:

SWL-OL-200 "Nitroaromatics and Nitroamines by HPLC," Method 8330
SWL-OL-201 "Polynuclear Aromatic Hydrocarbons (PAH) by HPLC"

8.5.4 Ion Chromatographs (IC) — The calibration procedure is found in SOP, SWL-IN-301, "Anions in Water by Ion Chromatography"

8.5.5 Mass Spectrometry Section — The calibration procedure are found in the following technical SOPs:

SWL-OV-303 "Volatile Organics by GC/MS (SW846, Method 8260B
SWL-OV-304 "Volatile Organics by GC/MS (SW846, Method 8260B, 25mL Purge)"
SWL-OV-311 "Volatile Organics — by GC/MS Low Concentration CLP"
SWL-OV-315 "Volatile Organics by 524.2, Drinking Water, 25 mL Purge"
SWL-OV-317 "Analysis of Purgeables, Method 624"
SWL-OV-318 "Volatile Organics by OLM04.2 — EPA — CLP"
SWL-OS-500 "Semivolatile Organics by SW846, Method 8270C"
SWL-OS-503 "Semivolatile Organics by CLP OLM03.2"
SWL-OS-504 "Semivolatile Organics in Low Concentration Water OLC2.1"
SWL-OS-506 "Semivolatile Organics in Air by GC/MS (TO-13)"
SWL-OS-507 "Analysis of Semivolatile Organics by EPA 625"
SWL-OS-509 "Semivolatile Organics by EPA CLP Method OLM04.2"

SWL-OD-202, "Polychlorinated Dibenzo-P-Dioxins and Dibenzofurans 8280A"
SWL-OD-203, "Polychlorinated Dibenzo-P-Dioxins and Polychlorinated Dibenzofurans (USEPA DFLM01.0)"
SWL-OD-204, "PCDD and PCDF by High Resolution Mass Spetroscopy (EPA 1613A/DLM01.2)"
SWL-OD-205, "PCDD and PCDF by High Resolution Mass Spetroscopy (SW846, Method 8290, and TO-9/EPA23)"
SWL-OA-101, "Volatile Organics in Air by TO-1"
SWL-OA-102, "Volatile Organics in Air by GC/MS (TO-14/TO-15)"
SWL-OA-103, "Analytical Modified Method TO-3 for the Determination of Volatile Organics in Air by GC/MS"

8.6 Balance Checks

Analytical balances of various capacities and operational mode are calibrated every six months, by a licensed specialist and officially recorded as verification of performance. Details of the calibration procedures are found in SOP SWL-IN-120, "Calibration of Electronic Balances".

Stickers documenting the calibration are placed on the balance noting the calibration date as well as the date of the next calibration. Certificates of calibration are maintained in the QA/QC department.

Balances are checked with known calibration weights before using. If the values of the calibration are not within limits, the balance will be calibrated per manufacturer's instructions.

8.7 Calibrating Thermometers

Thermometers are calibrated annually against an NIST thermometer using the following procedure:

- Install the NIST thermometer in the same environment as the thermometer in question (e. g. cold storage refrigerator, oven, etc.)
- Should the thermometer in the cold storage be in glycerin, verify that the NIST thermometer is in the same solution.
- Allow the thermometers to equilibrate.
- Read both thermometers and record in the calibration log.
- Verify that the thermometer in question is within the ±0.2 acceptance limit.
- Should the temperature be outside this limit, the thermometer should be replaced with a new calibrated thermometer.
- Flag thermometer with any applicable bias.

8.8 Calibration Verification Volumetric Labware

Labware used for sample measurement (i.e. pipettor, etc.) are checked each day of use as per ASTM instructions and checked gravimetrically every 6 months. Details of these checks are provided in SOPs SWL-IN-100 and SWL-OP-203.

8.9 Radiochemistry

The calibration procedures are found in the following SOPs:

- SWL-RD-117, "LB4100 Low Background Counting System"
- SWL-RD-119, "Alpha Spectrophotometer Operation/Calibration"
- SWL-RD-125, "Gamma Spectrophotometer Operation/Calibration"
- SWL-RD-131, "Liquid Scintillation Operation/Calibration"
- SWL-RD-135, "Sodium Iodide Detector Operation/Calibration"
- SWL-RD-136, "Survey Meters/Probes"

8.10 Documentation — See Appendix B on Logbook Maintenance Standard Operating Procedures.

9.0 DETECTION LIMITS

All MDLs will be determined annually for each instrument, method, matrix and analyte using SOP, SWL-GA-113, "Definition and Determination of Detection Limits". MDL studies will also be performed if the Section Supervisor determines that a significant change in the analytical process has occurred, or to allow the operator to demonstrate initial and continuing analytical

proficiency. The QAO, or his designee, must approve all MDL study results, and all associated reporting limits.

The value reported to clients as the lowest possible concentration is based on the reference method or client-specific requests. The value to be reported is documented in the project-specific QAP for client-specific requests as well as in the "Redbook". The Redbook lists the in-house, validated, lowest possible concentration as measured by the in-house test method. The MDL determined by SWLO should not exceed the MDL as defined in the reference Method. The Redbook lists the in-house method, revision number, date performed, the calculated MDL, and the reporting limit. The IDL, when applicable, is discussed in the appropriate in-house method.

In some reference methods, a process for determining the lowest possible concentration to be measured is not specified. The SWLO procedure found in SOP, SWL-GA-113, "Definition and Determination of Detection Limits", which is based on 40 CFR Part 136 for the determination of MDLs is followed when the reference method does not specify a mechanism for determining the lowest possible concentrations. The process for determining the lower limit to be reported by the laboratory when not specified by the client or the reference method is defined in the SWLO in-house method.

The reporting limit as presented on the laboratory reports as "detection limit" is defined by the in house analytical SOP. This limit may correspond to the PQL, CRQL, CRDL or other limits as appropriate for the reference method, client or contract. The use of IDLs, MDLs and PQLs and other contract required limit determinations (e. g. CRDLs or CRQLs) in reporting results are determined first by any specific customer contract requirements. If there are no contract-specific requirements, then the applicable reference method requirements for limit reporting are observed. Finally, if there are no method requirements for reporting, the reporting limits as defined in each SWL analytical SOP are observed. For contracts/projects requiring the use of non-EPA approved methods, the laboratory shall establish detection limits according to the contract requirements and protocol. Clients requiring matrix specific MDL studies using site specific matrices are performed using SWL-GA-113 as referenced above, or a documented client-specific procedure.

9.1 Reporting Limit

If a project requires a reporting limit less than the lowest calibration standard, then a check standard must be analyzed at that regulatory limit (unless the level is below the MDL.)

The reporting limit may be the method specified (e. g. the CRQL in CLP) or method defined practical quantitation limit as specified in the RCRA methods or other method defined sensitivity. In all cases the establishment of the reporting limit is documented in the in-house analytical SOPs. The reporting limit must never be less than the calculated Method Detection Limit from the in-house method as determined by the laboratory.

The reporting limit is always adjusted based on sample dilution, sample concentration or sample size. Client's developed or project's reporting limits based on project-specific requirements are documented in the client file and noted in the report to the client. In no case are client specific reporting limits, using the same in-house procedure, less than laboratory developed method detection limits.

10.0 QUALITY CONTROL CHECKS

10.1 Laboratory Control Checks

10.1.1 Method Blanks

Method blanks, also known as reagent blanks, are analyzed for each matrix with each batch of 20 or fewer samples. An aliquot of equal volume or weight to the sample is used for method blank analysis. The method blank, like that of duplicate and spike samples, is taken through the whole analytical process. The method blank shall not be contaminated with any analyte of interest above the reporting limit. In the case of volatiles and semivolatiles, contamination must be less than the reporting limit of common laboratory solvents or phthalates, except for EPA, CLP or client permission where the contamination can be up to 5X the reporting limit.

10.1.2 Calibration Blank/System Blank

A calibration blank/system blank is prepared by analyzing the same matrix used for the preparation of the calibration standards. It is used to establish the analytical curve by taking into account background responses during the calibration process. It is also used to check for carry-over contamination after a standard run or after a contaminated sample run.

10.1.3 Calibration Verification

Calibration Verifications are used to assure calibration accuracy during each analysis run. It must be analyzed for each analyte as described in the particular SOP. For inorganic analysis initial calibration verification (ICV) must be analyzed to verify the accuracy of the initial calibration. A continuing calibration verification (CCV) must be analyzed at the beginning of the analysis, after every ten samples, and at the end of the run. The ICV is prepared from an independent source different than the calibration standards. The CCV is prepared to be near or at the mid-range levels of the calibration curve. CCV's must also vary over the range of the calibration.

10.1.4 Laboratory Control Spikes (LCS)

Laboratory Control Spikes (LCS) are prepared from standards received from commercial vendors. These are a secondary source from those standards used to prepare calibration (either from another vendor or another lot from the same vendor). LCS are analyzed each day for every sample batch run to check proficiency of the analysis in terms of working standards preparation, monitor standard degradation, and check traceability of the prepared standards. A measure of comparability between batches is established with the analysis of the LCS. It is also used to check efficiency of both the digestion/extraction, and the instrumental analysis. An LCS duplicate (LCSD) may also be analyzed with each batch to check precision (except for air analyses).

The percent recovery is determined by the following equation:

$$\text{Percent Recovery} = \frac{\text{LCS Found Value}}{\text{LCS True Value}} \times 100$$

10.1.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

MS/MSD is used to assess the impact of the sample matrix on analyte recovery and precision. These are replicate samples spiked with a known spike concentration that are taken through the whole sample preparation process. MS/MSD analyses are performed on one sample in each group of 20 samples (5%), and/or on each type of sample matrix per concentration. Normally the client decides which sample(s) is/are to be analyzed as matrix spikes. In those instances where samples are not specified, the procedure is for the laboratory to randomly pick a sample (excluding field blanks, trip blanks, etc.) representative of the batch. Table 10.1 shows a list of matrix spike compounds routinely employed by the laboratory. If the client requires that all targeted analytes of interest be spiked, this will also be done for each MS/MSD. For inorganic analyses, all elements of interest are spiked.

The amount of spike added varies according to the working range of the analytical instrument and is at least 10X the MDL.

After the value of the sample is determined, the value of the sample spike is determined. The percent (%) recovery of the spike is calculated using the following equation:

$$\%\ \text{Recovery} = \frac{(\text{Spike Sample Result} - \text{Sample Result}) \times 100}{\text{Spike Added}}$$

At times the sample value is outside the linear range of the analytical method. In such cases it is impossible to know the magnitude of the analyte before the spike is added. Occasionally, the sample and the spike sample require dilution to perform sample analysis within the linear range of the instrument. This dilution adjusts the analyte in the sample to the proper concentration, but it will sometimes also dilute the spike added below a detectable recovery. In this case, it is not possible to report spike recovery for that particular analyte.

The lack of spike recovery data for an analyte that has been diluted to levels outside the working concentration of the instrument is supplemented by the periodic analysis of spiked QC check sample and other additional sample data.

The calculated % recoveries are then used to assess data precision expressed as relative percent deviation (RPD). It is calculated using the following equation:

$$\text{RPD} = \frac{(\text{MS Result} - \text{MSD Result}) \times 100}{\text{Mean of MS and MSD Results where MS denotes Matrix Spike.}}$$

10.1.6 Duplicate Analyses

RPD can also be measured by duplicate sample analysis of one sample. In this case the (2) sample results take the place of the MS and MSD values in the above equation. The procedure of choosing which sample to duplicate when unspecified by the client is the same as detailed above in Section 10.1.5.

10.1.7 Surrogate Spike Analyses

Where applicable (organic analysis), the analytical process includes the addition, subsequent detection, and recovery calculation of surrogate spiking compounds. Surrogate compounds are analyte compound substitutes, (i.e., compounds not specifically requested to be determined as analytes in a particular scope of work) which most often do not occur naturally. Surrogate compounds are added to samples for analysis and are taken through the whole sample preparation process. Surrogate compounds, to be useful in QC analysis, must not interfere with the determination of the analytes of interest. Surrogates must also be chemically similar to the analytes of interest and capable of emulating the analyte response.

Surrogate standard determinations are performed on all samples and blanks. All samples and blanks are spiked with surrogate spiking compounds before purging or extraction in order to monitor preparation and analysis of samples.

Table 10.2 shows a list of surrogate spike compounds routinely employed by the laboratory.

Acceptance criteria for surrogate recoveries are method dependent and are found in the Method SOPs.

10.1.8 Method of Standard Addition (MSA)

A method of standard addition is used to check the accuracy of the analysis method under optimum conditions, excluding chemical interference from sample matrix. The MSA procedure is performed for GFAA analysis in accordance with CLP protocol for all samples.

10.1.9 ICP Interference Check Sample (ICS) Analysis

ICP interference check sample verifies interelement and background correction factors of the ICP instrument. The ICS is analyzed at the beginning and at the end of the run, or every 8 hours, whichever is more frequent. Recoveries must be ± 20% of the true value or all samples in that run must be reanalyzed.

10.1.10 Internal Standards Analysis

Internal standard areas are monitored as a measure of instrument calibration. Internal standard determinations are performed on GC/MS analyses to monitor instrumental efficiency and also used as a reference retention time indicator to check retention time shifts of peaks of interest.

A known amount of internal standard concentration is added to a sample extract prior to instrumental analysis. Like the surrogate standard, it must not interfere with the determination of the analytes of interest. It must also be chemically similar to the analytes of interest and capable of emulating the analyte response.

10.1.11 Post-Digestion Spike

If the matrix spike is outside of method control limits for metal and cyanide analyses, an aliquot of the unspiked sample will be used to determine if matrix interference is the cause. The sample will be spiked with a concentration of either two times the reporting limit or two times the sample concentration, whichever is greater.

TABLE 10.1

List Of Matrix Spiking Standards for Organic Analysis (Unless Otherwise Requested by the Client)

Matrix Spikes

Method 601/602/8021B (GC Purgeables)
All Compounds of Interest

Method 625 (Semivolatiles)
All Compounds of Interest

Method 608/8081A/IFB (Pest/PCB)
All Pesticides

Method 8082 PCB
Aroclors 1016/1260

Method 8151A (Herbicides)
All Compounds of Interest

Method 8260B/624 (GC/MS Volatiles)
All Compounds of Interest

Method 8270C (Semivolatiles)

Base/Neutrals Acids

1,2,4-Trichlorobenzene
Acenaphthene
2,4-Dinitrotoluene
Pyrene
N-Nitroso-di-N-Propylamine
1,4-Dichlorobenzene

Phenol
Pentachlorophenol
4-Chloro-3-Methylphenol
2-Chlorophenol
4-Nitrophenol

TABLE 10.2

List Of Surrogates Spiking Standards For Organic Analysis

Surrogates

Method 601/602/8021B (GC Purgeables)
Bromochloromethane
Bromofluorobenzene

Method 608/8081A/IFB/8082 (Pest/PCB)
Tetrachloro-m-xylene (TCX)
Decachlorobiphenyl (DCB)

Method 624/8260B/IFB (GC/MS Purgeables)
1,2 Dichloroethane-d4
Bromofluorobenzene
Toluene-d8

Method 8260B (GC/MS Purgeables)
4-Bromoflurobenzene
Dibromofluoromethane
Toluene-d8

Method 625/8270C/IFB (Semivolatiles)
Nitrobenzene-d5
2-Fluorobiphenyl
2,4,6-Tribromo-phenol
Phenol-d5
2-Fluorophenol
Terphenyl-d14
2-Chlorophenol-d4*
1,2-Dichlorobenzene-d4*

Method 8151A (Herbicides)
2,4-Dichlorophenylacetic Acid (DCAA)

*Advisory Surrogates

10.2 Sample Batching

A batch is defined as a group of samples prepared at the same time and location, using the same method. At SWLO/AATS the number of samples in a batch will not exceed (a) twenty field samples or (b) the number of samples which can be ran simultaneously or in a continuous sequence without interruption, whichever is lower.

Each batch of samples will contain at a minimum, a method blank (PB), a laboratory control sample (LCS), a matrix spike (MS), and a matrix spike duplicate or duplicate. The acceptance criteria for these quality control samples are present in each Method SOP. All preparation batches are labeled with a unique identification. For example:

97 05 08 SB

where: YY MM DD M R

year month day matrix run

Each PB and LCS is labeled with an identifier to tie them to the preparation batch.

10.3 Data Quality Objectives

10.3.1 Introduction

The laboratory scope and approach to produce data of known and sufficient quality are described in this section. Guidelines are provided for the assessment and reporting of data quality for any environmentally related measurements, and for the incorporation of such assessments into major environmental databases.

Controlled sample receiving, logging, and tracking throughout the length of the project/contract is maintained to ensure sample integrity throughout the sample analysis scheme. Documentation of instrument performance and preventive maintenance is used to provide a permanent record for data validation. SWLO checks the quality of analytical work through analysis of quality control (QC) reference samples, duplicate samples, or matrix spike duplicate and spike samples.

10.3.2 Data Quality

Data quality is the totality of features and characteristics of data that bear on their ability to satisfy a given purpose. Parameters of major importance are accuracy, precision, completeness, representativeness, and comparability. These are defined as follows:

- *Accuracy* — The degree of the difference between measured or calculated values and true value.
- *Precision* — The reproducibility or degree of agreement among replicate measurements of the same quantity.
- *Completeness* — The percentage of valid data obtained from a measurement system.

- *Representativeness* — The degree to which the data accurately represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition.
- *Comparability* — The confidence with which one data set can be compared to another.

10.3.3 Accuracy

The accuracy of the measurement data is evaluated by the comparison of the percent recovery of the QC reference material of known or established concentration, independent of routine calibration. Statistically based control limits are established for each method of analysis and sample matrix (soil/water). A Laboratory Control Spike (LCS) is analyzed for each batch and is dependent upon the sample matrix, method of analysis and concentration level. Recoveries are assessed to determine method efficiency. Analytical accuracy is expressed as the percent of recovery of an analyte/parameter that has been added at a known concentration before preparation and analysis. The equation used to calculate percent recovery is as follows:

$$\text{Percent Recovery} = \frac{\text{LCS Result} \times 100}{\text{Amount of Spike Added}}$$

Accuracy can also be assessed on the recoveries of spiked analytes in a sample Matrix Spike.

$$\text{Percent Recovery} = \frac{(\text{Matrix Spike Result} - \text{Sample Result}) \times 100}{\text{Amount of Spike Added}}$$

10.3.4 Precision

The laboratory uses Laboratory Control Spike Duplicates, Matrix Spike Duplicates or duplicate sample analysis to assess precision. An LCS/LCSD, MS/MSD, or a duplicate sample are analyzed for each batch of 20 samples (5%) for in-house QC and are dependent upon the sample matrix and method of analysis. A more frequent analysis is performed (i.e., one in 10 samples) on a reference method or contract-specific basis. The basic precision statistics obtained from the multiple batch frequency are compared to develop a graph assessment (using control limits) for given sample matrix.

Analytical precision is expressed as a percentage of the difference between the results of two matrix spike samples or two duplicate sample analysis for a given analyte. Relative percent difference (RPD) is calculated as follows:

$$\text{RPD} = \frac{(\text{LCS Result} - \text{LCS Duplicate Result}) \times 100}{\text{Mean of LCS and LCS Duplicate Results}}$$

Where: LCS denotes Laboratory Control Spike.

$$RPD = \frac{(\text{MS Result - MS Duplicate Result}) \times 100}{\text{Mean of MS and MS Duplicate Results}}$$

Where: MS denotes Matrix Spike.

...Or ...

$$RPD = \frac{\text{Sample Result - Duplicate Result}}{\text{Mean of Sample and Duplicate Results}}$$

10.3.5 Completeness

For the data to be valid, it must meet all the acceptance criteria including accuracy, precision and any other criteria specified by the analytical method used. Data validation procedures are employed to minimize the amount of bad data from getting through data collection.

While the quality objective is to obtain the greatest accuracy and precision, the specific accuracy/precision level is dependent on the method of analysis and type of sample matrix. The Methods SOP statistical control limits are used as guidelines to validate the data generated unless client or contract requirements set more stringent criteria.

10.3.6 Representativeness

Data generated by the laboratory shall be representative of the samples received and analyzed by the laboratory. It shall be representative of the laboratory database of accuracy and precision measurements of the particular parameter(s), matrix and analytical method.

10.3.7 Comparability

Data shall be comparable to data sets recorded in the past to check for historical consistency. In order to maximize its usefulness, data shall be reported in appropriate units and in a consistent manner and following the reference method. Data shall be reproducible under similar conditions whether generated by the laboratory or another firm. This is demonstrated through the use of inter-laboratory performance audit samples (e. g. WP, WS).

10.4 Method Validation

All analysts must be certified for proficiency in each test method they performed. Details of the proficiency testing program are found in SOP, SWL-GA-104, "Personnel Training".

10.5 Statistical Evaluation of the Data

10.5.1 Control Charts

The performance of a measurement system will be demonstrated by the

measurement of homogeneous and stable control samples in a planned repetitive process. The data generated is plotted in control charts to indicate whether the system is in control. It warns the laboratory of possible deviation from 95% confidence level by identifying systematic errors, drifts, or other types of problems. The control chart mechanism is how SWLO develops internal statistical limits. The discussion on the use and generation of control charts are found in the SOP, SWL-GA-112, "Control Charts".

The use of control charts is summarized as follows:

- They provide graphic assessment of accuracy and precision for the analysis of each analyte and instant detection of erroneous data
- They allow efficient observation of recovery trends for any particular analysis.
- They provide a long-term mechanism for self-evaluation of analytical output.
- They provide assessment of the analytical capability of the staff chemists with regard to the output of valid analytical data.
- They allow observations of deviations from control trends.

10.5.2 Control charts are generated annually for laboratory control samples and quarterly for MS/MSD samples. Automated control charts are generated for Volatiles, Semivolatiles, Pesticides/PCBs, Metals and Cyanide. Manual control charts are generated in the Radiochemistry, HPLC, Inorganics, and air analyses.

Control Chart Analysis

The following actions will require immediate corrective action:

- One measurement exceeds the control limit.
- Two successive points exceed the warning limit.
- Seven successive points on one side of the mean.
- Six successive points are in decreasing or increasing order.

11.0 OUT-OF-CONTROL EVENTS

11.1 An "out of control" event is defined as any occurrence failing to meet the Laboratory QA Plan, SOP, Reference Method or client specifications.

11.2 Criteria Used for Determination of an "out of control" Event:

Factors that affect data quality (failure to meet calibration criteria, inadequate record keeping, improper storage, or preservation of samples) require investigation and corrective actions.

11.3 Responding to an "out of control" Event

11.3.1 Roles and Responsibilities:

When an "out of control" event is recognized, each individual involved with the analysis in question has an interactive role and responsibility. Some examples are as follows:

- Sample Receiving
  If a sample is broken upon receipt, the Project Officer/Client is notified and the decision is documented.

- The Analyst
  He/she must be able to recognize QC failure and immediately notify the Laboratory Supervisor and work with the QAO to solve the problem.

- The Laboratory Supervisor
  He/she must review all analytical and QC data for reasonableness, accuracy, and clerical errors; also responsible to monitor QC charts (in terms of control limits). In an "out of control" event, the Laboratory Operations Manager works with the analyst and QAO to solve the problem. Stopping work on the analysis prevents the reporting of suspect data and ensures that all results that are suspect are repeated, if possible, after the source of the error is determined and remedied.

- QA Officer
  In the event an "out of control" situation occurs that is unnoticed at the bench or supervisory level (e. g. performance failure on a QC sample), the QAO will notify the Laboratory Operations Manager. The QAO will help identify and solve the problem where applicable, and ensure that work is stopped on the analysis and no suspect data is reported.

- Project Officer
  The Project Officer will immediately notify the client when project or method requirements are not met. The Project Officer will discuss possible consequences of not meeting project requirements with the client.

11.4 Procedures for Stopping Analysis

Whenever the analytical system is "out of control", investigation/ correction efforts are initiated by all concerned personnel.

If the problem is instrumental or specific only to preparation of that sample batch, samples analyzed during the "out of control" event are processed after the instrument is repaired and re-calibrated, provided holding times are not exceeded.

If a sample batch is still "out of control" after reanalysis, all method-related activities shall stop immediately. A detailed laboratory-wide investigation shall be conducted to isolate and correct faulty operations. Sample security, integrity of standards, reagents, glassware, laboratory notebooks, instrument performance and adherence to the methods shall be included in the investigation following the guidelines in SOP SWL-GA-108, "Performance and System Audits" and/or SOP SWL-GA-105, "Corrective Action".

All actions shall be documented and placed in their respective case/contract file as well as the Corrective Action file. Upon correction, departmental follow-up, and QA follow-up the corrective action can be closed out with "return to control" (see SWL-GA-105).

11.5 QC Reports

Results from QC are evaluated at the analyst level. Blank contamination and Laboratory Control Spikes outside of control limits warrant the re-preparation/analysis of the sample batch. Surrogate spikes are also evaluated to determine whether there were adequate recoveries from the extraction process. The method blanks and spikes are reported to the client. Internal standard recoveries are evaluated to assure that they meet the requirements of the SOP.

12.0 DATA PROCESSING

12.1 Laboratory Data Recording

All analysts/technicians shall document all information relating to the conduct of analytical test methods and related activities in bound laboratory notebooks. These notebooks serve as the primary record for subsequent data reduction. "Stand alone" computers generate the data for GC/MS, AA, ICP, Mercury and GC analyses. Details of maintaining these log books can be found in a series of SOPs listed in Appendix B. Following the review by the Analyst, Group Leader and/or supervisor, the data is downloaded from the instrument to the department's database. The results for other analyses are transcribed manually onto "Analytical Results" forms specific to the analysis.

12.2 Data Review

The review of data quality involves several levels of evaluation. In general, the analysts and the Program Manager are responsible for reviewing the data relative to instrument calibration, standard preparation, method blanks, raw data, calculations and transcriptions. The analyst reviews 100% of the raw analytical data generated, including the calibration data and all calculations. Upon completion of the analysis process, a

100% peer review is performed. Group Leaders/Section Supervisors perform a second level of review of the raw data. At this level, a 100% review for compliance is performed for method, QC, and Project requirements.

12.2.1 A third level of review involves the Program Managers or Project Officer[8]. This is generally the final review prior to the QA Officer's review.

12.2.2 At the next level of data quality review, the QA Officer is generally responsible for a review of a minimum of 10% of the data generated. The emphasis is on the data acceptability relative to the data quality indicators and on the accuracy of the final data summaries.

12.2.3 Review Checklists

The review checklist and review procedures are found in the department data review SOPs: SWL-OV-103, "Volatile Data Review"; SWL-OS-303, "Semivolatile Data Review"; SWL-OP-100, "Gas Chromatography Data Review"; SWL-IN-106, "1st and 2nd Review of ICP, GFAA, Mercury, and Cyanide"; SWL-IN-107, "1st and 2nd Review of Wet Lab Analysis".

12.3 Data Collection/Data Reduction

Following the reviews by the analyst and the group leader and/or supervisor, the data is downloaded from the instrument to the departmental database. Data clerks transfer the results into the software report forms. The data is then qualified and organized in accordance with the following SOPs: SWL-GA-132, "Data Reporting — Non-EPA"; SWL-IN-102, "Inorganic Data Package Assembly"; SWL-OS-405, "GC/MS Data Package Generation with Enviroquant Software"; SWL-OV-106, SOP for Volatile Data Clerks; SWL-OS-300, "Organic Data Package Assembly"; SWL-OP-300, "Enviroquant File Management"

Data packages are then prepared. The Supervisor and/or Program Manager then reviews the data packages.

12.3.1 Gas Chromatograph Results

Calculations are performed for each analyte after it is identified. Identification is based on analyte peaks falling within established retention time windows on both the primary and secondary (confirmation) columns. The concentration of the analyte is determined by using the calibration curve and the peak area of the analyte. A response factor is determined from the calibration curve and used to calculate the concentration.

12.3.2 Mass Spectrometry/Gas Chromatograph Results

Qualitative identifications are determined by obtaining extracted ion current profiles (EICPs) for the primary ion mass to charge ratio (m/z) and the secondary masses for each analyte. Positive identification is based on the following criteria:

[8] This review assures that all required levels of laboratory review have been performed and that project requirements have been met. Problems with the analysis, deviations from methods, and/or client communications are reviewed and addressed in the case narrative.

- The retention time must fall within ± 30 seconds of the retention time of the daily calibration standard.
- The relative peak heights of the characteristic masses in the EICPs must fall within ± 20% of the relative intensities of these masses in a reference mass spectrum (standard analysis or reference library).

If a compound cannot be verified by all of the criteria above, yet in the technical judgement of the mass spectral interpretation specialist the identification is correct, the compound shall be reported.

The calculation for the concentration for the suspect peak is made using the RF for each analyte.

$$\text{Concentration} = \frac{(A_s)(C_{is})}{(A_{is})(RF)}$$

Where:

A_s = Area of characteristic m/z for the analyte to be measured.

A_{is} = Area of characteristic m/z for the internal standard

C_{is} = Concentration of the internal standard, in ug/L.

RF = Average response factor as calculated from the area formed on an intensity plot of the ion of interest.

12.3.3 Inductively Coupled Plasma

The purpose of the ICP is to measure concentrations of elements in a sample. This is accomplished by measuring the emission intensity produced when a sample containing these elements is aspirated into the plasma, exciting the electrons to a higher state. The measurement of intensity is performed by allowing the light leaving the plasma through each exit slit to fall on a photomultiplier tube.. This tube converts light energy into an electrical current. At the end of the integration time, the total current produced is measured and is proportional to the intensity and the concentration of the element being analyzed. This total current value is sent to the computer for data reduction. The final calculations are done by the computer system by comparing the intensity of unknowns against the intensity of known standards.

Interelement correction is performed by measuring the light intensity of interfering elements and mathematically correcting for the additional emitted intensity.

12.3.4 Atomic Absorption Spectrophotometry

Photometric absorbance is governed by the relationship:

$$\text{Absorbance} = \log(100 / \%T) = 2 - \log \%T$$

Where:

% T = 100 - % absorption

Percent absorption is based on the amount of light of a particular wavelength absorbed by a specific metal. Its calculation is based on the loss of light of a particular wavelength as it is passed through a flame into which a solution containing metals of interest have been aspirated.

Calibration curves establishing the absorption relationship with concentration are generated at various concentrations. From these curves, a comparison is made with absorbance from the sample measurement. Since absorbance is directly related to concentration, a plot of the two parameters is linear in certain operable ranges and allows for determination of unknown concentrations in solutions (direct samples or extracts) after measurement of absorbance.

Atomic absorption spectrophotometry is based on the principle that when light of a resonance wavelength is passed through a graphite tube containing atoms of an element to be measured, part of the light is absorbed. The extent of the absorption is proportional to the number of atoms present in the atomized sample. Because of the sophistication of current instrumentation, the partial application of this technique for the measurement of metal concentration in liquids relies on a Beer's absorption law approach, comparing absorbance from an unknown against the linear correlation between absorbance and concentration in standards.

In many spectrophotometric measurements, interferences occur in the absorption of light confusing the Beer's law relationship between absorption and concentration. This is especially true for atomic absorption. To alleviate this problem, a technique known as the "method of standard additions" is used, whereby sequential, known amounts of the component for which the sample is being analyzed are added to a sample of unknown concentration. By making an initial absorbance measurement after each addition, for which the effects of interference present in the analytical matrix can be accounted.

12.4 Data Verification

The Project Officer and/or the Quality Control Officer then verify the data.

- Review of client chain-of-custody request vs. sample login.
- Review of Extraction Logs, Analytical Run Logs, etc.
- Review of performance indicators such as blanks, surrogate recoveries, LCS recoveries, and Matrix Spike results.
- Random calculation checks
- Review and approval of the final report.

12.5 Data Reporting

The data reporting process provides the final product to the clients. Hardcopy and/or Electronic Data Deliverables (EDD) are sent; details regarding report formats are included in the following SOPs: SWL-GA-132 "Data Reporting — Non-EPA"; SWL-IN-102, "Inorganic Data Package Assembly"; SWL-OS-301, "Organic Data Package Assembly", SWL-OS-405, "GC/MS Data Package Generation with Enviroquant

Software"; SWL-OV-106, SOP for Volatile Data Clerks; SWL-OP-300, Enviroquant File Management.

13.0 HOLDING TIMES AND PRESERVATIVES

Table 13.1 and 13.2 list the recommended preservatives and holding times for the various analytes/parameters. When samples arrive, sampling dates are entered into the LIMS system upon log-in. The project officer is notified if a holding time might be exceeded and the project officer in turn notifies the client. A daily LIMS list is printed in the various sections detailing these parameters and samples approaching holding time deadlines (Fig. 13.3). This enables the laboratory personnel to set priorities to meet holding time requirements. In the event that holding times are exceeded, the Project Officer will notify the Field Sampling Manager or client and request a resampling or instructions for proceeding with the analysis. Details for samples outside of holding times are stated in the case narrative with the final report.

Table 13.1

Recommendation For Sampling And Preservation Of Samples According To Measurement(1)

Measurement	Vol. Req. (mL)	Holding Container[2]	Preservative[3,4]	Time[5]
Physical Properties				
Color	50	P, G	Cool, 4°C	48 Hrs.
Conductance	100	P, G	Cool, 4°C	28 days
Hardness	100	P	HNO_3 to pH <2	6 mos.
Odor	200	G only	Cool, 4°C	24 Hrs.
pH	2	P, G	None Required,	Analyze Immediately
Residue				
Filterable	100	P, G	Cool, 4°C	7 days
Non-Filterable	100	P, G	Cool, 4°C	7 days
Total	100	P, G	Cool, 4°C	7 days
Volatile	100	P, G	Cool, 4°C	7 days
Settleable Matter	1,000	P, G	Cool, 4°C	48 Hrs.
Temperature	1,000	P, G	None Req.	Analyze Immediately
Turbidity	100	P, G	Cool, 4°C	48 Hrs.
Metals				
Dissolved	200	P	Filter on site HNO_3 to pH <2	6 Mos.
Suspended	200		Filter on site	6 Mos.[8]
Total	100	P	HNO_3 to pH <2	6 Mos.
Chromium^{+6}	200	P	Cool, 4°C	24 Hrs.
Mercury (Dissolved)	100	P	Filter HNO_3 to pH <2	28 Days
Mercury (Total)	100	P	HNO_3 to pH <2	28 Days

(cont.)

Table 13.1

Recommendation For Sampling And Preservation Of Samples According To Measurement[1]

Measurement	Vol. Req. (mL)	Holding Container[2]	Preservative[3,4]	Time[5]
Inorganics, Non-Metallics				
Acidity	100	P, G	Cool, 4°C	14 Days
Alkalinity	100	P, G	Cool, 4°C	14 Days
Bromide	100	P, G	None Req.	28 Days
Chloride	50	P, G	None Req.	28 Days
Chlorine	200	P, G	None Req.	Analyze Immediately
Cyanides	500	P, G	Cool, 4°C NaOH to pH >12 0.6g ascorbic acid[6]	14 Days[7]
Fluoride	300	P, G	None Req.	28 Days
Iodide	100	P, G	Cool, 4°C	24 Hrs.
Sulfate	50	P, G	Cool, 4°C	28 Days
Silica	50	P only	Cool, 4°C	28 Days
Sulfide	500	P, G	Cool, 4°C add 2 mL zinc acetate plus NaOH to pH >9	7 Days
Sulfite	50	P, G	None Req.	Analyze Immediately
Nitrogen				
Ammonia	400	P, G	Cool, 4°C H_2SO_4 to pH <2	28 Days
Kjeldahl, Total	500	P, G	Cool, 4°C H_2SO_4 to pH <2	28 Days
Nitrate plus Nitrite	100	P, G	Cool, 4°C H_2SO_4 to pH <2	28 Days
Nitrate[9]	100	P, G	Cool, 4°C	48 Hrs.
Nitrite	50	P, G	Cool, 4°C	48 Hrs.
Dissolved Oxygen				
Probe	300	G bottle and top	None Req.	Analyze Immediately
Winkler	300	G bottle and top	Fix on site and store in dark	8 Hrs.

(cont.)

Table 13.1

Recommendation For Sampling And Preservation Of Samples According To Measurement[1]

Measurement	Vol. Req. (mL)	Holding Container[2]	Preservative[3,4]	Time[5]
Phosphorus				
Orthophosphate, Dissolved	50	P,G	Filter on site Cool, 4°C	48 Hrs.
Hydrolyzable	50	P,G	Cool, 4°C H_2SO_4 to pH <2	28 Days
Total	50	P,G	Cool, 4°C H_2SO_4 to pH <2	28 Days
Total, Dissolved	50	P,G	Filter on site	24 Hrs.
Organics				
BOD	1,000	P,G	Cool, 4°C	48 Hrs.
COD	50	P,G	Cool, 4°C H_2SO_4 to pH <2	28 Days
Oil & Grease	1,000	G only	Cool, 4°C H_2SO_4 or HCl to pH <2	28 Days
Organic Carbon	25	P,G	Cool, 4°C H_2SO_4 or HCl to pH <2	28 Days
Phenolics	500	G only	Cool, 4°C H_2SO_4 or HCl to pH <2	28 Days
MBAS	250	P,G	Cool, 4°C	48 Hrs.
NTA	50	P,G	Cool, 4°C	24 Hrs.
Purgeable Halocarbons (601)	40	G, Teflon-lined Septum	Cool, 4°C 0.008% $Na_2S_2O_3$[6]	14 Days
Purgeable Aromatic Hydrocarbons (602)	40	G, Teflon-lined Septum	Cool, 4°C, 0.008% $Na_2S_2O_3$[6], HCl to pH <2	10 Days[10]

(cont.)

Table 13.1

Recommendation For Sampling And Preservation Of Samples According To Measurement[1]

Measurement	Vol. Req. (mL)	Container[2]	Holding Preservative[3,4]	Time[5]
Organics (continued)				
Acrylonitrile & Acrolein (603)	40	G, Teflon-lined Septum	Cool, 4°C, 0.008% $Na_2S_2O_3$[6], Adjust pH to 4-5[11]	14 Days
Phenols[12] EPA Method 604	1,000	G, Teflon-lined Cap	Cool, 4°C, 0.008% $Na_2S_2O_3$	7 Days until extraction, 40 days after extraction
Benzidines[12] EPA Method 605	1,000	G, Teflon-lined Cap	Cool, 4°C, 0.008% $Na_2S_2O_3$[6], H_2SO_4 to pH 2-7[13]	7 days until extraction
Phthalate Esters[12] EPA Method 606	1,000	G, Teflon-lined Cap	Cool, 4°C	7 days until extraction, 40 days after extraction
Nitrosamines[12] EPA Method 607	1,000	G, Teflon-lined Cap	Cool, 4°C, store in dark, 0.008% $Na_2S_2O_3$[6]	7 days until extraction, 40 days after extraction
Organochlorine Pesticides/PCBs[12] EPA Method 608	1,000	G, Teflon-lined Cap	Cool, 4°C, pH 5-9	7 days until extraction, 40 days after extraction
8081A/8082 Nitroaromatics and Isophorone[12] EPA Method 609	1,000	G, Teflon-lined Cap	Cool, 4°C, 0.008% $Na_2S_2O_3$[6], store in dark	7 days until extraction, 40 days after extraction
Polynuclear Aromatic Hydrocarbons[12] EPA Method 610/8310	1,000	G, Teflon-lined Cap	Cool, 4°C, 0.008% $Na_2S_2O_3$[6] store in dark	7 days until extraction, 40 days after extraction

(cont.)

Table 13.1

Recommendation For Sampling And Preservation Of Samples According To Measurement[1]

Measurement	(mL)	Vol .Req. Container[2]	Holding Preservative[3,4]	Time[5]
Organics (continued)				
Haloethers[12] EPA Method 611	1,000	G, Teflon-lined Cap	Cool, 4°C, 0.008% $Na_2S_2O_3$	7 days until extraction, 40 days after extraction
Chlorinated Hydrocarbon[12] (612)	1,000	G, Teflon-lined Cap	Cool, 4°C	7 days until extraction, 40 days after extraction
TCDD[12] EPA Method 1613 8280A/8290	1,000	G, Teflon-lined Cap	Cool, 4°C, 0.008% $Na_2S_2O_3$[6]	7 days until extraction, 40 days after extraction
Purgeables EPA Method 624/601/602 8021B/8260B	3 x 40	G, Teflon-lined Septum	Cool, 4°C, HCl 0.008% $Na_2S_2O_3$[6]	14 Days
Base/Neutrals Acids EPA Method 625/8270C	1,000	G, Teflon-lined Cap	Cool, 4°C, 0.008% $Na_2S_2O_3$[6]	7 days until extraction, 40 days after extraction
Radiochemistry				
Tritium	200	Glass	None	6 months
All others	1000	P,G	HNO_3 to pH < 2	6 months

Footnotes:

1 More specific instructions for preservation and sampling are found with procedures as detailed in EPA-600/4-79-020, revised March, 1983, and in the Federal Register, Vol. 49, No. 209, Oct. 26, 1984, EPA 40 CFR part 136.

2 Plastic (P) or glass (G). Only EPA certified pre-cleaned bottles are used. For metals, polyethylene with a polypropylene cap (no liner) is preferred.

3 Sample preservation should be performed immediately upon sample collection. For composite samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then samples may be preserved by maintaining them at 4° C until compositing and sample splitting is completed.

4 When any sample is to be shipped by common carrier or sent through the US Mail, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements of Table 1, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: (1) Hydrochloric Acid (HCl) in water solutions at concentrations of % by weight or less (pH about 1.96 or greater). (2) Nitric Acid (HNO_3) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater). (3) Sodium Hydroxide (NaOH) in water solutions at concentrations of 0.08 by weight or less (pH about 12.3 or less).

5 Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still considered valid. Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that the specific types of sample under study are stable for the longer time and has received a variance from the Regional Administrator. Some samples may not be stable for the maximum time period given in the table. A permittee, or monitoring laboratory, is obligated to hold the sample for a shorter time if knowledge exists to show that this is necessary to maintain sample stability.

6 Should only be used in the presence of residual chlorine.

7 Maximum holding time is 24 hours when sulfide is present; Optionally, all samples may be tested with lead acetate paper before the pH adjustment in order to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.

8 Samples should be filtered immediately on-site before adding preservatives for dissolved metals.

9 For samples from non-chlorinated drinking water supplies concentrated H_2SO_4 should be added to lower sample pH to less than 2. The sample should be analyzed before 14 days. Samples receiving no pH adjustment must be analyzed within seven days of sampling.

10 The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of the sampling.

11 When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4° C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6 – 9. Samples preserved in this manner may be held for 7 days before extraction and 40 days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 6 (requirement for thiosulfate reduction of residual chlorine). .

12 If 1,2-Diphenylhydrazine is likely to be present, adjust the sample pH to 4.0 ± 0.2 to prevent rearrangement to benzidine.

TABLE 13.2

Recommendation for Sampling and Preservation of Soil Samples According To Measurement[A]

Measurement	Container Size (oz)	Container	Holding Preservative	Time
Inorganics, Non-Metallics				
Petroleum Hydrocarbons	32	G	Cool, 4°C	28 days
Metals				
Total Recoverable	8	G	Cool, 4°C	6 months
Mercury (total)	8	G	Cool, 4°C	28 days
Extraction Procedure Toxicity	32	G	Cool, 4°C	6 months
Organics				
VOCs and Xylenes, EPA Method 8260B	2 x 4	G	Cool, 4°C	14 days
Encore	3 x 2	Teflon	Cool, 4°C	48 hrs[B]./14 days
Extractable Priority Pollutants EPA Method 8270C	32	G	Cool, 4°C	14 days – extraction, 40 days – analysis

A More specific instructions for preservation and sampling are found with procedures as detailed in SW846, "Test methods for Evaluating Solid Waste, Physical/Chemical Methods", 3rd Edition, Chapter 4.

B To preservation.

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GA-100 Part 3 of 3

The Standard Operating Procedures (SOPs) contained herein are adopted by Pace Analytical Services.

These SOPs will be followed until superceded by Pace Analytical Services SOPs.

Signature:	Signature:	Signature:
Chuck Hoover Quality Assurance Officer Pace Analytical Services, Inc. Broken Arrow, Oklahoma	Roger C. Splinter Dir. of Quality, Safety, Training Pace Analytical Services, Inc. Minneaplolis, MN 55414	Dave Neal General Manager Pace Analytical Services, Inc. Lenexa, KS

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

Standard Operating Procedure

Laboratory Quality Assurance Plan

Document No.: SWL-GA-100

Rev No. / Date: Rev. 10.1 — 08/11/03

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APPROVALS	DATES
Melissa Chance Procedure Prepared By	8-11-03 Date
Robert Mathis Laboratory Manager or Director	8/14/03 Date
Chuck Hoover Laboratory Safety Officer	8/11/03 Date
Chuck Hoover Laboratory QA/QC Officer	8/11/03 Date

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

Document Status

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Figure 13.3

Holding Time Report

SOUTHWEST LABORATORY OF OKLAHOMA, INC.
Department Week Report — Department : MASS SPECTROMETRY — Date : 04-25-1997 — Page : 1
Test : MS3XX — Report : Holding Times — Start date : 04-16-1997

Wed 04/16/97	Thur 04/17/97	Fri 04/18/97	Sat 04/19/97	Sun 04/20/97	Mon 04/21/97	Tues 04/22/97

Test code: MS300 VOLATILE ORGANICS POL TABLE 2 — S: 10 days W: 10 days O: 10 days

S 29077.01 B /1	S 29081.01 B /1		W 29077.13 N			
S 29077.02 B /1	S 29081.02 B /1		S 29080.01 B			
S 29077.03 B /1	S 29081.03 B /1		S 29080.02 B			
S 29077.04 B /1	S 29081.04 B /1		S 29080.03 B			
S 29077.05 B /1	S 29081.05 B /1		S 29080.04 B			
S 29077.06 B /1	S 29081.06 B /1		S 29080.05 B			
S 29077.07 B /1	S 29081.07 B /1		S 29080.06 B			
S 29077.08 B /1	S 29081.08 B /1		S 29080.07 B			
S 29077.09 B /1	S 29081.09 B /1		S 29080.08 B			
S 29077.10 B /1	S 29081.10 B /1		S 29080.09 B			
S 29077.11 N /1			W 29080.10 B			
S 29077.12 B /1						
S: 12	S: 10		S: 9			
W: 0	W: 0		W: 2			
O: 0	O: 0		O: 0			

Test code: MS317 VOLATILES-EPA CLP OLM03.1 — S: 10 days W: 10 days O: 10 days

		S 29067.01 C			
		S 29067.02 C			
		S 29067.03 C			
		S 29067.04 C			
		S 29067.05 C			
		S 29067.06 C			
		S 29067.07 C			
		S 29067.08 C			
		S 29067.09 C			
		S 29067.10 C			
		S 29067.11 C			
		S 29067.12 C			
		S: 12			
		W: 0			
		O: 0			

Test code: MS399 VOA BY GC/MS MOBSTANDARD — S: 10 days W: 10 days O: 10 days

S 29040.01 C		S 29070.01 C			
S 29040.02 C		S 29070.02 C			
S 29040.03 C		S 29070.03 C			
S 29040.04 C		S 29070.04 C			
S 29040.05 C		S 29070.05 C			
S 29040.06 C		S 29070.06 C			
S 29040.07 C					
W 29040.16 C					
S: 7		S: 6			
W: 1		W: 0			
O: 0		O: 0			

S: 19	S: 10	S: 10	S: 9
W: 1	W: 0	W: 0	W: 2
O: 0	O: 0	O: 0	O: 0

14.0 QUALITY ASSURANCE AUDITS/EXTERNAL AGENCY APPROVAL

14.1 Audit is defined as systematic check to determine the quality of operation of laboratory activities. It is comprised of the following:

- Performance audit
- System audit

14.1.1 Laboratory Performance Audit

Procedures used to assess the effectiveness of the quality control system are as follows:

- Internal Performance Audits.

  Internal performance audits are accomplished through the QAO who monitors that the requirements of the LQAP and SOPs are being met. The use of control samples, replicate measurements and reference materials

also help to monitor the system's performance. Internal audits and yearly assessments are conducted as per SOP, SWL-GA-108, "Performance and Systems Audits". Performance Evaluation Samples are analyzed as typical samples without replicate testing.

Sample analysis systems for each laboratory area are conducted by the QA Officer and include the following:

- Verification of written procedures and analyst(s) understanding.
- Verification and documentation of procedures and documents.
- Review of analytical data and calculations.

The internal technical audits evaluate compliance with the QA Plan, the SOP and with method and client requirements. Internal technical audits (as well as system audits) are conducted as per a predetermined schedule and/or as per managerial request. Internal audit findings are tracked using the corrective action process discussed in SOP SWL-GA-105, "Laboratory Corrective Action". With the predetermined schedule the entire technical operation of the lab is reviewed annually.

14.1.2 Laboratory System Audit

Internal Systems Audits are performed by laboratory quality assurance personnel. The inspection reviews the laboratory quality control system including sample handling, sample analysis, record control, preventive maintenance, and proficiency testing. As with the technical audits, the system audits are conducted as per a predetermined schedule and/or as per managerial request. SWLO's entire Quality System is evaluated annually. Non-Conformance and/or Corrective Actions Identified are tracked as discussed in the SOP SWL-GA-105, "*Laboratory Corrective Action*".

14.1.3 Blind QC Program

Intra-laboratory check samples are analyzed on a quarterly basis for a full suite of organic and inorganic parameters. These samples are entered into the system as "blind" QC samples. Also, "blind" QC samples may be entered into the system in conjunction with external performance audits, or to check analyst proficiency. When the analysis is complete, laboratory performance is evaluated and any necessary corrective action is taken. The QAO monitors all results and maintains all QC performance sample data. The special processing of these blind QC checks and all PE samples is forbidden.

14.1.4 External Performance Audits

External performance audits are accomplished through inter-laboratory checks such as:

- Participation in various state laboratory evaluation programs.
- Participation in WP & WS studies from NSI/RTC and Accustandard.
- Analysis of split samples and comparing results with the other laboratory.
- Participation in the U.S. EPA CLP program.

- Participation in the U.S. Army Corps of Engineers DERA validation program.
- Participation in the U.S. Navy NFESC Program.

14.1.5 Audit Review

All audit and review findings and any corrective actions that arise from them shall be documented. The laboratory management shall ensure that these actions are discharged within the agreed time frame.

14.2 External Certifications and Approvals

The SWLO/AATS laboratory has participated in available certification programs pertaining to environmental chemistry. SWLO/AATS participates in numerous certification/approval programs (both state and federal). The federal programs are listed in Table 14.1 and the state programs are listed in Table 14.2.

TABLE 14.1

Program Participation

USEPA CLP (Contract Laboratory Program)

- Organics Analysis, Multi-Media, Multi Concentration, by GC/MS and GC/EC Techniques. *Note: Continuous Participation Since 1985.*
- Rapid Turn-Around Organics Analysis, Multi-Media, Multi Concentration, By GC/MS And GC/EC Techniques.
- Chemical Analytical Services For Multi-Media, Multi-Concentration Metals And Inorganics.

Environmental Resource Associates/Analytical Products Group/Accustandard

- WP/WS Water Pollution/Water Supply Study Program.
- Soil P. T. — Studies/RAD Chem Studies
- Accu Standard WS • Studies (Dioxin)

U.S. Army Corps Of Engineers DERP Certification (Defense Environmental Restoration Program).

U.S. Air Force AFCEE IRP, Installation Restoration Program Participant, Analytical Services For AFB Projects.

U.S. Navy NFESC, Installation Restoration Program Participant, Analytical Services.

INEL, (Idaho National Engineering Lab.), Qualified By ERPSMO To Perform Analytical Services.

NRC License, Licensed To Perform Analysis Of Environmental Samples. (License #35-27413-01)

US Department of Energy, Environmental Management Consolidated Audit Program

TABLE 14.2

State Certification*

State	Certification Number	Department	Type	Expires	Evaluation Process
ARIZONA	AZ0436	Environmental Lab, Dept. of Health	ENV, HW		On-Site, WP
CALIFORNIA	1221	Department Of Health Services, Environmental Lab Program	ENV		Reciprocity (NELAC)
COLORADO	335	Department of Health, Drinking Water Parameters	DW	12/31/03	Reciprocity (OK)
FLORIDA	87326	Department of Health and Rehabilitation	DW,ENV	06/30/04	On-Site, WP
FLORIDA	87376	Department of Health and Rehabilitation	DW,ENV	06/30/04	On-Site, WP
FLORIDA	87530	Department of Health and Rehabilitation	DW,ENV	06/30/04	On-Site, WP
GEORGIA	922	Environmental Protection Division	ENV		Reciprocity (FL NELAC)
IDAHO	—	Department of Health and Welfare	DW	08/31/03	Reciprocity (OK)
KANSAS	E-10155	Department of Health and Environment	HW	10/31/03	Reciprocity (NELAC)
KANSAS	E-10155	Department of Health and Environment	DW	10/31/03	Reciprocity (NELAC)
KANSAS	E-10155	Department of Health and Environment	WW	10/31/03	Reciprocity (NELAC)
KENTUCKY	90065	Department of Environmental Protection	DW	12/31/03	Reciprocity (OK)
LOUISIANA	93-19	Department of Health and Hospitals	ENV	06/30/04	Reciprocity (NELAC)
MARYLAND	274	Department of Health	DW	09/30/03	Reciprocity (OK)
NEW YORK	11672	Department of Health	ENV	04/01/04	Reciprocity (NELAC)
OKLAHOMA	8728	Water Resources Board	DW, ENV	08/31/04	On-Site, WS/WP
OREGON	OK-200001	Oregon ELAP	DW, ENV		Reciprocity (NELAC)
PENNSYLVANIA	EP-LAB-16	Department of Environmental Protection	DW	02/01/04	On-Site (WS)
SOUTH CAROLINA	79003	Department of Health and Environmental Control	HW, ENV	09/24/03	Reciprocity (OK)
TENNESSEE	02929	Department of Health	DW,UST	02/21/05	Reciprocity (OK)
UTAH	SWLO1	Department of Health	ENV, HW	06/30/04	Reciprocity (NELAC)
WASHINGTON	N/A	Department of Health	UST,DW	09/05/03	Reciprocity (COE)
VIRGINIA		Department of General Services	DW	06/30/04	Reciprocity (OK)

* Certificates and certified parameter listings can be found in the GP-Series SOPS, QA Department, and/or Marketing Certification Records.

15.0 REFERENCES

- Naval Facilities Engineering Science Center, Interim Guidance Document, "Navy Installation Restoration Chemical Data Manual", September 1999.
- Department of Defense Quality Systems Manual for Environmental Laboratories, Version 1, October 2000.
- National Environmental Laboratory Accreditation Conference, :Quality Systems", Revision 4, January 3, 1997.
- Office of Nuclear Safety Policy and Standards, DOE Order 5700.6C: "Quality Assurance Program, A Management System, Revision 1, October, 1992.
- USEPA SW846, "Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods", Revision 2, September, 1994, and Update III, December, 1996.
- USEPA Contract Laboratory Program, "Statement of Work for Organic Analysis, Multimedia, Multi-concentration", OLM03.2 & OLM04.2.
- USEPA 40CFR 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.
- USEPA Contract Laboratory Program, "Statement of Work for Inorganic Analysis, Multimedia, Multi-concentration", ILM04.0.
- USEPA "Methods for the Determination of Metals in Environmental Samples", EPA 600-91-010, June, 1991.
- USEPA "Methods for Chemical Analysis of Water and Wastes", EPA 600/4-79-020.
- "Standard Methods for the Examination of Water and Wastewater, 18th Edition.
- Air Force Center for Environmental Excellence, "Quality Assurance Project Plan", ver. 2.0.
- US Army Corp of Engineers, "Shell for Chemical Analytical Services – version 1.0, 2Nov98.
- Department of Defense Systems Manual for Environmental Laboratories – version 1, Oct. 2000.
- US Department of Energy Environmental Management Consolidated Audit Program.
- US Department of Energy, Office of Environmental Management, Quality Systems Manual for Analytical Services, July 2002, Draft Rev. B.

16.0 DOCUMENT CONTROL AND STORAGE

Detailed instructions and guidelines for the preparation, review, approval and distribution control of documents affecting the quality and consistency of the work performed by SWLO/AATS are found in SOP, SWL-GA-101, "Preparation, Review, Revision and Control of Procedural Documents". Written documents are prepared and approved for all activities affecting the quality and consistency of work performed by SWLO/AATS. Activities include, and 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; general laboratory practices which support these activities. In addition, procedures are written which conform to government regulations affecting worker health and safety, hazardous waste management, and radiation protection. These procedures are governed by the same controls as those affecting quality of work.

The policies and practices for the archival and retrieval of raw data are detailed in SOP, SWL-GA-106, "Archival and Retrieval of Sample Documentation". Policies and practices for electronic media are found in SOP, SWL-GA-102, "Information Systems Quality Assurance Plan". All systems are designed so that data, records, and documents can be stored and retrieved ten years after the date of generation.

In the case that the laboratory is sold, the purchasing company would be interested in being assigned existing contracts and maintaining existing certifications. It would be a condition of sale that all contract requirements, including storage and archiving of data, would be assigned to the new corporation.

In the event of the laboratory being forced to close, the likelihood of long-term storage of archived data would be remote. The plan would be to review the current archived records of data and contact those clients with existing data and give them a 60-day time period to either have the data shipped to them or have the data destroyed based upon their requirements.

17.0 MATERIALS AND SUBCONTRACTORS

The policies, procedures and practices for the procuring and approving of materials and equipment are detailed in SOP, SWL-GA-107, "Procurement of Materials and Equipment". Policies for the verification of the quality of standards are located in each departments' "Standards and Traceability" SOPs.

17.1 Procurement and Inventory

17.1.1 Southwest Laboratory of Oklahoma, Inc. has established a system to assure that products and services, purchased or contracted, shall meet at least the minimum standards required. Factors such as cost, volume of work, ease of operation, inherent accuracy, expected equipment lifetime, length and condition of warranties or service contracts, expected downtime and repair costs are considered during the selection process. The increased usage of electronic analytical instruments has improved the quality and quantity of data and has increased productivity.

17.1.2 Control of materials (e. g. reagents, standards, solvents) and glassware used in the analyses is maintained as part of the quality assurance program. Reagents and solvents are analyzed prior to use to verify purity, documentation of these analyses are maintained. Lot numbers are recorded on preparation logs to facilitate the tracking of these items. This will correlate the analytical reagent lot number to specific analytical batches. All reagents are dated as they are received and when they are opened to assure systematic use. The identity, purity, shelf-life, source, tests to be considered for quality and purity, storage and handling procedures, and replacement dates are factors that are considered in making purchase requisitions.

Before any purchases are made, the purchase requisition orders and requests are checked and verified by the appropriate laboratory supervisor. The authority for approval for such purchases is held by the laboratory supervisors and the Laboratory Director.

17.2 Equipment Management

17.2.1 Information on performance of the equipment is obtained before a purchase request is made. Service availability for installation specification and verification is considered in purchase negotiation. When the instrument/equipment is installed, an internal calibration is made on the instrument to meet manufacturers' and Reference Methods' specifications. Calibration checks are done by using analytical reference standards for qualitative and quantitative checks to verify instrument performance during the sample run. Routine preventive maintenance of the instruments or equipment is made on a regular basis. Section 8.0 discusses preventive maintenance employed by the different laboratory sections to ensure instrument/equipment working conditions.

17.3 Supplies Management

17.3.1 Materials, reagents, solvents and gases are carefully selected to meet specifications as prescribed by the method of analyses. Each new supply of these items is verified for performance capability based on the required certified assay/analysis of chemicals and freedom from impurities that would interfere with the analysis. Background levels are measured to check the degree of contamination through conformance of storage requirements according to the manufacturer's directions and/or individual method of analysis. Solvents used for extraction are pre-analyzed to determine impurities that might interfere with the analytes of interest. Standards and reagents are dated, initialed and labeled with the expiration date (if not manufacturer stated) upon receipt. This procedure establishes the order of use and eliminates the possibility of exceeding shelf life.

17.4 Source of Standard Reagents

17.4.1 Primary standards and/or stock standards are obtained from a reliable, certifiable source and are high purity. Standards are purchased from approved commercial vendors such as Chem Services, Fisher Scientific, Supelco, etc., for use in all analytical testing. Standards are protected from degradation, deterioration and contamination based on storage requirements (e.g. polyethylene containers for

alkali solutions, glass containers for organics and brown glass for light-sensitive solutions: temperature storage and segregation of standards based on reactivity).

Stock and working standard solutions are prepared fresh as required by their stability, and are checked regularly for signs of deterioration, (e. g. discoloration, formation of precipitates, and changes in concentration). Standard solutions are labeled with lot number, expiration date, preparation date and preparer.

17.5 Glassware

17.5.1 Class A volumetric glassware is used by the laboratory for accurate measurements in both inorganic and organic analysis.

Laboratory contamination is minimized through implementation of a standard operating procedure (SOP) for glassware and labware cleaning. It is followed to ensure the removal of all traces of parameters of interest and contaminants that could interfere with analysis.

17.6 Reagents, Solvents and Gases

17.6.1 Chemical Reagents aside from the primary standard reagents, solvents and gases are carefully selected to conform to specifications defined in the method SOP. Selection is based on the required purity for parameters being measured, sensitivity of the method and specificity of the detection system (e. g. AA, ICAP, GC-ED, GC/MS).

17.6.2 Laboratory reagents obtained from approved commercial vendors shall meet AC standards and are labeled indicating contents, date of receipt or preparation, and expiration date (where appropriate). Hazardous reagents are adequately labeled and stored segregated from the rest of the reagents to indicate type and degree of hazard. All solvents are dated upon receipt, and again upon opening, to ensure "first in – first out" usage. Solvent bottles are stored in a grounded flammable liquid storage cabinet.

17.6.3 Gases used in organic and inorganic analyses are of commercial grade or are laboratory-supplied gases. For organic analyses, the type of detection (e. g. GC-EC, Hall, GC-FID, GC/MS) used affects gas quality requirement. Molecular sieves, carrier-gas filters, and drying tubes are required on combustion gases to improve quality. Gas cylinders are immediately replaced when the pressure falls to 100-200 pounds per square inch (psi) to minimize detector contamination that will affect sensitivity of the detector.

17.7 Laboratory Reagent Water

ASTM Type II water (by conductivity) is used in the laboratory for dilution, preparation of reagent solutions and final rinsing of glassware while ASTM Type I water (by resistivity) is used for metals analysis. It is free from interferences and other contaminants. After passing through two ion exchange canisters and one carbon filter canister, conductivity is monitored and recorded daily and an indicator light, which (when out) alerts the analyst to have the tanks serviced.

17.8 Compressed Air

Compressed air is employed mainly in instruments using GC oven-door control and autosamplers. Absorption filters are installed between the outlets and the point of use to trap oil, moisture, and other contaminants entering the compressed air transfer lines. These lines are checked for the presence of moisture and contaminants and are replaced as soon as moisture is detected.

17.9 Hood System

An efficient hood system is necessary to remove the various toxic and hazardous fumes that may be generated when using organic solvents or that may be formed during an acid digestion step. It is also used to remove toxic gases that may be formed during atomic absorption analysis reactions. The laboratory fume hood face velocity is regularly checked every four months for optimum face velocity.

17.10 Electrical Services

The laboratory electrical system provides adequate and constant voltage, appropriate grounding, and efficient lighting which is required for satisfactory lighting, proper functioning of sensitive instruments and operation of high-current devices. A licensed electrical contractor provides repairs and services in the event of a power failure or electrical problems.

17.11 Subcontractors

In the event that subcontracted services are required, the procurement process shall be governed by SWLO's "Small Business Contracting Plan." The plan includes a historical list of small businesses that have performed to specifications in the past. The plan also includes the utilization of sources provided by the Small Business Administration (SBA). The current plan is maintained by the laboratory's manager of the Contracts/Proposal Department. It is the responsibility of the Contracts/Proposals Manager to write a solicitation that includes all of the 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 is not limited to the following:

- Any required state and/or federal agency certifications, validations, and/or proof of the successful completion of Performance Evaluation (PE) samples and on-site audits.
- The applicable scope of work and quality assurance objections from the Prime Contractor'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 SWLO's Prime Contractor (see SWL-GA-107.)

18.0 QA REPORTS TO MANAGEMENT

18.1 Quality Assurance Reports to Management

Weekly a Quality Assurance Report is produced for Management. This report covers

results from internal and external audits, performance evaluation samples, data reviews, training, proficiency, certification, document control, non-conformances and corrective action issues. Figures 18.1 & 18.2 re examples of the weekly quality systems reports.

18.2 Managerial Review

The laboratory management conducts a weekly review of the quality system to ensure its continuing suitability and effectiveness and to introduce any necessary changes or improvements in the quality system and laboratory operations. The review shall take account of reports from QA, managerial and supervisory personnel, the outcome of recent internal audits, assessments by external bodies, the results of inter-laboratory comparisons or proficiency tests, any changes in the volume and type of work undertaken, feedback from clients, corrective actions and other relevant factors. More details of this review can be found in the Standard Operating Procedure SWL-GA-142, "*Management Assessment of the Quality Systems.*"

Figure 18.1

Weekly Staff Meeting Report

QA DEPT.
WEEKLY STAFF-MEETING REPORT
FOR WK/ENDING 04/28/2000

A. DATA REVIEW

1. NO DATA REVIEWED.

B. TRAINING/PROFICIENCY

1. TABULATING PROFICIENCIES IN S.C. FORMAT ALONG WITH NELAP CERT. STATEMENTS.
2. TRAINING FILE REVIEW - SEVERAL MISSING ANNUAL QA PLAN TRAINING.

C. CONTROL CHARTS

1. '99 CHARTS NEEDED FOR 2000 LIMITS.

D. COMPLAINTS/CORRECTIVE ACTIONS/NON CONFORMANCES

COMPLAINTS

1. No Complaints Noted.

CORRECTIVE ACTIONS/NON CONFORMANCES

1. See Audit Issues

DATA RESUBMISSIONS

1. See Attached.

CORR. ACTNS/NON CONF'S REPORTS NEEDED

1. See Audit Issues

FOLLOW UPS NEEDED

1. '99 unclosed C.A.s and Non-Conformances

E. LABORATORY QA PLAN

1. QAP Under Revision.

F. AUDITS SCHEDULED

EXTERNAL AUDITS

1. NO AUDITS SCHEDULED

INTERNAL AUDITS

1. VOA's
2. HAZWASTE - Keith
3. MIS -Rob

PLANNED FOR MAY

1. INORGANICS
2. RESPIRATORY PROTECTION PLAN
3. CONTROL CHARTS
4. ZONE BALANCE CHECKS

(cont) **Figure 18.1**

Weekly Staff Meeting Report

QA DEPT.
WEEKLY STAFF-MEETING REPORT

FOR WK/ENDING 04/28/2000

(CONTINUED)

PAGE 2

G. AUDIT RESPONSES

EXTERNAL

1. INEL -FOLLOW-UP (GALP ISSUES)
2. KERRM -OUT
3. RMA
4. OKLA. ENV. (see Attached)- OUT
5. ARIZ. - Due June 2nd.
6. EPA
7. FLORIDA-NELAP

INTERNAL	Responsible	Suggested Date
1. **Smpl Rec. -** Navy Aqueous Smpl Storage		
2. **MIS**	Action Items	
3. **P.O.s**		
Initial Contact Forms	*Marketing*	???
Non-Standard Requirements w/ ARF & Wrksheet		
Communication Form	*P.O.s*	
Project Files to Central Location	*P.O.s*	
4. **SVOAs**		

H. EXTERNAL PEs

IN-HOUSE	DATE RECEIVED	DATE DUE
1. **N.C. (42509 & 42602)**	**04/11/2000**	**05/01/2000**
2. **NELAP SOILS (42803)**	**04/27/2000**	**5/17/00**
3. **ADDITIONAL NELAP PEs? –SEE ATTACHED**		

PEs SENT OUT / AWAITING GRADING

	DATE SENT
1. MAPEP	04/14/2000

I. INTERNAL PEs

	DATE RECEIVED	DATE DUE
NONE		

J. MISC. SUPPORT

1. SEVERAL SOPs SENT TO TOM JOHNSTON TETRATECH-NUS.
2. HELPED D. LEMASTERS PUT TOGETHER TRAINING DOCUMENT REGARDING SPECIAL PROJECT REQUIREMENTS - FOR PROPOSAL.

K. SAFETY

1. **EXTRACTIONS HOOD $ QUOTE TO BE REC'D.**

Figure 18.2

QC Tracking Coversheet

QC Tracking Coversheet

I. Overdue SOP's

II. Two-month SOP Outlook

III. Outdated Redbook Entries

IV. In-House PE Samples

Appendix A-1

Personnel Experience

Key Personnel Resumes

ROBERT HARRIS

Laboratory Director

EDUCATION: Bachelor of Science Degree, Microbiology
Oklahoma State University, 1971

PROFESSIONAL EXPERIENCE:

7/1/83 to Present — **Southwest Laboratory of Oklahoma, Inc.**
Laboratory Directors
As the Laboratory Director of Southwest Laboratory of Oklahoma Mr. Harris directs all aspects of laboratory operation in the Company's facility, including scheduling and cost control, staffing, training, customer support, and business development. He holds a B.S. in Microbiology from Oklahoma State University and has over 15 years experience in management of commercial laboratories. Analytical services under his direction include hazardous waste, drinking water and ground water investigation sponsored by a wide variety of governmental and private clients including EPA (e.g.,CLP,RCRA) Corps of Engineers (DERA) and the Air force (OHEL/HSD).

Prior to joining SWL he managed a geochemisty laboratory for Williams Brothers Engineering which supported major oil companies. Mr.Harris has developed supplemental methods for the analysis of hazardous waste when there were no applicable ones available for specific project requirements. His experience in directing the operations of environmental laboratories is exemplified by successful participation in the EPA CLP, U.S. Army Corps of Engineers, and other federal and State certification and accreditation programs. As an analyst Mr. Harris is experienced in the application of analytical methods using SW846, CLP, ASTM, and Standard Methods for organic and inorganic samples.

1981 to 1983 — **Williams Brothers Engineering**, Tulsa, Oklahoma
Assistant Laboratory Director
1975 to 1981 — Senior Research Engineer

1971 to 1975 — **Kansas City Testing Laboratory**, Kansas City, Missouri
Laboratory Manager

Appendix A-2

Personnel Experience

STEVE MARKHAM

Operations Manager & Inorganic Program Manager

EDUCATION: Bachelor of Science Degree, Zoology
Oklahoma State University, Stillwater, OK, May 1977.

Fisheries Biology
Frostburg State College, Frostburg, Maryland, 1979.

Additional Education:

Atomic Absorption Training Course, Perkin-Elmer
Class II Wastewater Treatment Operators License, Virginia (1979)
Inductively Coupled Argon Plasma Course, Perkin-Elmer
Project Officer's Training - US Environmental Protection Agency.
ICAP-61 Training Course - Thermo Jarrell Ash

PROFESSIONAL EXPERIENCE:

- As the Operations Manager for Southwest Laboratory of Oklahoma, Mr. Markham assists the Laboratory Director in all aspects of laboratory operations, including scheduling, staffing, training and quality assurance.
- Mr. Markham is responsible for assuring that the laboratory has the personnel and support to deliver data of the highest quality and integrity.
- Mr Markham has eighteen years experience in the environmental profession and twelve years of management experience.

PROFESSIONAL EXPERIENCE:

1996 to Present — **Southwest Laboratory of Oklahoma, Inc.**
Operations Manager

1994 to 1996 — **Gulf States Analytical, Inc.**, Houston, Texas
Operations Manager

7/88 to 1993 — **Southwest Laboratory of Oklahoma, Inc.**
Inorganic Program Manager

1986 to 1988 — **Weston Corporation**, Stockton, California
Inorganics Section Manager.

1985 to 1986 — **U.S. Environmental Protection Agency, (Reg.III)**, Annapolis, Maryland
Environmental Scientist.

1983 to 1985 — **Water Pollution Control**, Arlington County, Virginia
Chief Chemist.

1979 to 1983 — **Lapteff Associates**, Woodbridge, Virginia

Appendix A-3

Personnel Experience

JAYANT SHRINGARPURE

Laboratory Technical Director

EDUCATION: Ph.D., Organic Chemistry
Indian Institute of Technology, Bombay, India

Master of Science, Environmental Management
University of San Francisco

Master of Science, Organic Chemistry
Indian Institute of Technology, Bombay, India.

Bachelor of Science, Chemistry
Bombay University, Bombay, India

PROJECT EXAMPLES:

- Project Manager for USEPA. Contract involving the rapid response (16-hour electronic data transmission) for the analyses of dioxins up to 100 samples per day by GC/MS.
- Program Manager for USEPA-CLP Organic Contract at SWLO.

PROFESSIONAL EXPERIENCE:

1985 to Present — **Southwest Laboratory of Oklahoma, Inc.**
Technical Director
Responsible for supervising the operations of the Extractions and GC/MS laboratories, including troubleshooting of instrument difficulties and final data review for GC/MS. Involved in ongoing work with the Research and Development laboratory conducting method developments and validation studies. Acts as client liaison on projects involving technical difficulties.

1985 to 1992 — **Organics Program Manager**
Program Manager for the organic department at SWL. Supervises work assignments, conducts method development and method validation. Supervises the operation of extraction GC and GC/MS Laboratory. Trains analysts on the use of instrumentation as well as trouble-shooting problems with the instruments. Reviews data for the GC/MS Laboratory.

1980 to 1985 — **EAL Corporation**
Supervisor — Responsible for the development of analytical methods for the analysis of organic compounds in the environment.

1978 to 1980 — **Columbia University**, New York
Research Associate — Specialized in the syntheses and purification of retinoids used against epithelial cancer.

1976 to 1978 — **Fordham University**, New York
Research Associate — Investigated intramolecular imino Diels-Adler reaction for the syntheses of idolizidine alkaloids.

1975 to 1976 — **Bakul Chemicals**
Senior Chemist

1969 to 1975 — **Indian Institute of Technology**, India
Research Chemist

Appendix A-4

Personnel Experience

CHUCK HOOVER
Quality Assurance Officer

EDUCATION: Bachelor of Arts Degree in Biology
Minor in Chemistry
Wichita State University, 1976

PROJECT

- Experience with PCB analyses on transformer oils, Hydraulic fluids, soil/sediment, waters and fish flesh.
- Experience in EPA 600 series Methodologies, both extractions and analyses.
- Experience in Trihalomethane Studies on source and drinking water supplies.
- Experience in data review of analyses performed on samples from EPA's Superfund Sites; performed in various laboratories in the EPA's Contract Laboratory Program.
- Metal analysis using Flame/Furnace Atomic Absorption. Extraction/Clean-up of Dioxin/Furans using method 613.

PROFESSIONAL EXPERIENCE:

1987 to Present — **Southwest Laboratory of Oklahoma, Inc.**
QA/QC Officer

1985-1987 — **Lockheed Engineering and Management Services,** Las Vegas, Nevada
Technical Support Supervisor — Laboratory Performance Monitoring Section.

1983-1985 — **Southwest Laboratory of Oklahoma, Inc.**
QA/QC Officer — Extractions Chemist — AA Operator

1982-1983 — **National Analytical Laboratories**, Tulsa, OK
Gas Chromatography/GC/MS Chemist

1980-1982 — **Williams Brothers Laboratories**, Tulsa, OK
Wet Chemistry Section Supervisor

1977-1980 — **Wichita Water Department, Water and Waste-water Laboratory**, Wichita, KS
Laboratory Technician

1976-Summer — **U.S. Department of Labor Occupational Health & Safety Administration (OSHA),** Kansas City, MO
Industrial Hygienist (Summer Merit)

Appendix A-5

Personnel Experience

DIANA HOKE
Organic Program Mgr.

EDUCATION: Bachelor of Arts Degree, Chemistry
Miami University, Oxford, Ohio, 1985

Additional Courses:

RTE-A Advanced Operator Course, HP Training Center, Atlanta, GA, 02/91
RTE-A Operator Course, HP Training Center, Atlanta, GA, 05/89
5970B Operator Course #18952Q, HP Training Center, Naperville, IL 11/86

PROFESSIONAL EXPERIENCE:

8/92 to Present — **Southwest Laboratory of Oklahoma, Inc.**
Organic Program Manager — Responsible for day to day operations in the Organic department. Oversees performance for all GC and GC/MS operations including extractions, analyses, data review, and data management. Provides technical management for organic EPA contracts. Interviews prospects for organic personnel.

GC/MS Data Reviewer — Reviews all GC/MS semivolatile data, troubleshoots data problems. Also performs analysis of GC/MS semivolatiles.

11/88 to 8/92 — **Hayden Environmental Group, Inc.**
Organics Laboratory Supervisor — Initial setup of GC/MS system to accommodate EPA approved methods for analysis of volatile priority pollutants following EPA protocol. Initial setup of second system for semivolatile analysis. Responsible for operation and maintenance, methods development, troubleshooting instruments, and training additional operators. Scheduling work activities of five technicians. Operation and maintenance of GC/ECD, GC/FID, and GC/PID for the analysis of Pesticides/PCBs, herbicides, organic solvent identification, and other procedures in compliance with EPA and NIOSH methods. Certified by OEPA for volatile organic compounds in drinking water.

8/85 to 11/88 — **Environmental Enterprises, Inc.**
10/87 to 11/88 **GC/MS Laboratory Supervisor** — Training and supervision of two technicians in the operation and maintenance of GC/MS systems. Obtained OEPA Certification for the analysis of potable water.

3/86 to 10/87 **GC/MS Operator** — Analysis of volatile and semivolatile priority pollutants.

2/86 to 3/86 **GC Operator** — Analysis of Pesticides/PCBs.

8/85 to 2/86 **Lab Technician** — Performed wet chemistry analysis. Obtained OEPA certification for analysis of fluorides, chlorides, pH, phosphates, and alkalinity.

Appendix A-6

Personnel Experience

DESMOND FOSTER

Extractions Laboratory Supervisor

EDUCATION: Tulsa Junior College — coursework includes Chemistry and Biology
Open Bible Institute, Trinidad — one year general coursework
Art Diploma — London University, London England
Basic Lab Skills Diploma — Hess Oil, U.S. Virgin Islands
Diploma — Trinidad High School (1964)

PROFESSIONAL EXPERIENCE:

1981 to Present — **Southwest Laboratory of Oklahoma, Inc.**
1988 to present — **Organic Extractions Laboratory Supervisor,**

Organic Extractions (12 years)
Supervision of 13 chemists in extractions of multiple matrices for organic chemical analyses. Methods used include the U.S. EPA SW-846 3500 Series, the U.S. EPA Contract Laboratory Program (CLP) Organic Statement of Work (SOW 3/90 & 2/88). Explosives, Herbicides, High Concentrations, Phenols and TPH's. Polychlorinated dibenzo-p-dioxins/dibenzofurans (PCDD/PCDF) extractions using 8280 and CLP Rapid Turnaround SOW. Multiple clean-up techniques including the Columbia Fishery (for Dioxins/Furans) and GPC (for semivolatiles and pesticides/PCBs).

Inorganics (5 years)
Wet chemistry work consisted of colorimetric and titrimetric procedures of chloride, fluoride, nitrate, nitrite, sulfates, COD, ammonia, and TKN. Other tests were pH, conductance, total dissolved solids, total solids, total suspended solids, hardness, acidity, and TOC.

Gas Chromatography (5 years)
Natural Gas Analysis

Field Sampling (5 years)
Sampled soils, monitoring water wells, and lysimeters.

Stack Sampling (2 years)
Collected SOX, NOX and particulate samples.

Hazardous Waste Supervisor
Maintained records and prepared reports as per 40CFR part 262. Filled out manifests and coordinated pick-up of drums by hazardous waste firms - ENSCO and APTUS. Inspected drum staging area to ensure proper storage and labeling of drums.

1979 to 1981 — **Hess Oil Company**, St. Croix, Virgin Islands
Petroleum Analyst — Gauged and sampled tanks, ships and refinery units for crude, JP4, JP54, gasoline, kerosene, benzene, toluene, xylene, napthas, #2 oil, #4 oil, #6 oil, etc. Laboratory tests performed were flash point (open cup and closed cup), cloud point, pour point, freeze point, sodium chloride in crude, D86 distillation, D286 distillation, D1160 distillation, true boiling point distillation, particulates in jet fuel, fuel system icing inhibitor, Reid Vapor Pressure, color ASTM, saybolt and Viscosities (SUS, SFS and kinematic).

Appendix A-7

Personnel Experience

MARK SMITH

Information Systems Manager

EDUCATION: Masters of Biomedical Science Program
Major: Pharmacology, 25 credit hrs.
Oral Roberts University, Tulsa, Oklahoma, 1985

Bachelor of Science, Chemistry
Minor, Computer Science
Oral Roberts University, Tulsa, Oklahoma, 1984

PROFESSIONAL EXPERIENCE:

9/91 to Present — **Southwest Laboratory of Oklahoma, Inc.**

11/96 to Present — **Information Systems Manager**
Direct the in-house hardware and software development computer personnel. Develop programs to automate data reduction, reporting in CLP or CLP-like format, and the transfer of data electronically within the laboratory. Develop programs to generate client requested electronic data deliverables. System administrator for the HP-UX UNIX systems.

9/91 to 11/96 — **Organics Data Manager**
Develop software programs and UNIX scripts to assist in the data processing for the GC/MS and GC sections of the laboratory. Train users on data reduction software. Review Organic data packages. System administrator for the HP-UX UNIX systems.

9/89 to 9/91 — **NET, Inc.**, Carrolton, Texas
GC/MS Supervisor
Responsible for the scheduling and analysis of all samples for volatiles and semivolatiles by GC/MS. Responsible for the operation and maintenance of four HP GC/MS systems. Duties include data reviews, standards preparation, methods development, SOP development, writing software automation software, employee training, and instrument operation.

3/86 to 9/89 — **Southwest Laboratory of Oklahoma, Inc.**

11/88 – 9/89 — **Volatile Organics Group Leader**
Directed the operations of the volatiles GC/MS laboratory. Responsible for the scheduling and analysis of samples, so that holding time deadlines and turnaround times were met. Review data for quality and completeness. Responsible for the maintenance of the GC/MS systems.

8/86 to 11/88 — **GC/MS Chemist**
Analyzed environmental samples for volatiles, semivolatiles, and dioxins in accordance with EPA CLP or SW846 Methods.

3/86 to 8/86 — **Extractions and Inorganics Chemist**
Prepared extracts for GC and GC/MS analysis. Performed analysis for total organic carbon, oil and grease, chemical oxidation demand, and ion chromatography.

Appendix A-8

Personnel Experience

KERT SURFACE

Nuclear Chemistry Manager/Project Officer

EDUCATION: B.S. Geology, Minor in Computer Science
Stephen F. Austin University, 1987

PROFESSIONAL EXPERIENCE:

3/95 to Present — **Southwest Laboratory of Oklahoma, Inc.**
Nuclear Chemistry Manager, Radiation Safety Officer, Project Officer
Manages Radiochemistry laboratory with emphasis on instrumentation and wet chemical separations. Oversees sample receipt screening for mixed waste samples. Responsible for all activities including sample flow, production, data compilation for data deliverables, and chemical analysis for naturally occurring and man-made radionuclides. Trains chemists and technicians. Maintains second-line quality checks and turnaround time assurance in the radiochemistry laboratory and enforces all operating procedures, as well as US Department of Energy's requirements and client-specific requirements. Monitors control charts and sample preparation documents. Conducts technical audit tours in radiochemistry and login/sample receiving departments.

1/95 to 3/95 — **Texas A & M University**
Radiochemistry Manager, Geochemical Environmental Research. Completed radiation safety training seminars.

1990 to 1995 — **SCIENTECH, Inc.**
1992 to 1995 — Radiochemistry Manager, Developed and/or adapted standard analysis procedures for radiochemistry. Developed transuranics procedure for the chemical separation of americium and plutonium with routine yields of 60 to 100 percent, respectively. Developed working standard operating procedures for the analyses and instrumentation in the radiochemistry laboratory.

1990 to 1992 — Safety Officer, Radiochemistry Group Leader, Developed safety manual for environmental laboratory.

1989 to 1990 — **Alpha Energy Laboratories, Inc.**
Radiochemist — Duties includes radiochemistry, information management, and quality control. Developed computerized radiochemistry data reporting format under US Department of Energy's "General Radiochemistry and Routine Analytical Services Protocol" for data package assembling and electronic deliverables, as well as other client specific protocols.

Appendix A-9

Personnel Experience

ANN WADE
GC/MS Operator/Group Leader

EDUCATION: Bachelor of Science, Environmental Health
Indiana State University, Terre Haute, Indiana, 1986

Additional Courses:

RTE-A GC/MS Operator Course, Analytical Ed. Center, Atlanta, GA, 10/90

Health & Safety Supervisors Courses, Psara Technologies, 48 hours, 6/90

Building Inspection & Mgmt. Planning, Univ. of Illinois at Chicago, 2/88

Sampling & Analysis of Airborne Asbestos, NIOSH, Cincinnati, OH, 7/87

PROFESSIONAL EXPERIENCE:

7/92 to Present — **Southwest Laboratory of Oklahoma, Inc.**
GC/MS Operator/Group Leader
Conduct organic analysis of semivolatile environmental samples using combined gas chromatography/mass spectrometry (GC/MS) following EPA/CLP protocol and previously determined computer format. Prepare internal standards and calibration standards according to contract specifications. Check data from runs to ensure it meets protocol requirements. Assemble initial reports. Perform instrument maintenance to assure instrument is operating at maximum efficiency.

11/87 to 7/92 — **Hayden Environmental Group, Inc.**, Miamisburg, Ohio
Senior Organics Laboratory Technician

12/90 to 7/92 — GC Laboratory Group Leader/Technician, Equipment and instrument operation and maintenance, training of additional operators, and GC/MS analysis. Certified by OEPA for volatile organic compounds, insecticides and herbicides for drinking water.

5/90 to 12/90 — GC/MS Analyst, Operation, maintenance, of an RTE-A 5970 GC/MS system for volatile and semivolatile priority pollutant analysis and interpretation. Utilized EPA methods and CLP QA/QC protocol. Identify and evaluate health and safety hazards as Health & Safety Coordinator. Disposal of hazardous waste as Hazardous Waste Manager.

5/88 to 5/90 — GC Operator, Analysis of Pesticides/PCBs, solvent identification of industrial hygiene samples. Sample preparation, analysis, data reduction, reporting of results, and maintenance of instrument.

11/87 to 5/88 — Industrial Hygiene Technician, Perform contractor surveillance and asbestos sampling at abatement sites. Asbestos analysis by polarized light microscopy.

1984 to 1987 — **Internship Experience**

7/87 to 9/87 — **Environmental Specialist**—Franklin Envir. Testing, Inc.,
3/87 to 7/87 — **Environmentalist**—Warren Cty. Combined Health Dist.
5/85 to 8/85 — **Environmental Intern**—Warren Cty. Combined Health Dist.
5/84 to 8/84 — **Sanitarian Intern**—Dekalb County Health Department

Internship experience included asbestos air monitoring/sampling; noise/ventilation surveys; analysis of organics and metals; water sampling; and health inspections.

Appendix A-18

Personnel Experience
(Technical Personnel Experience Summary)

	Total Years Experience												
	Total Experience In Laboratory Field	Current Position	GC/MS	Purge & Trap	Gas Chromatography	High Performance Liquid Chromatography	Sample Preparation	Data Review	Inductively Coupled Plasma	Inorganic Analysis	Atomic Absorption	Data Reporting	Nuclear Chemistry
Assefa, Senait Extractions Technician M.S. Biochemistry	4	1					1						
Bolding, Blaine GC/MS Analyst B.S. Biology	3	2	2				2						
Brown, Shauna Analyst B.S. Biology	1	1											
Cannon, Autumn Inorganic Lab Technicians High School	2	4mo					2				4mo		
Crovador, Eliana Laboratory Technician H.S. Diploma	1.5	1.5											
Crovador, Maria Extractions Technician H.S Diploma	3	3					3						
Derouen, Chris Extraction Technician H.S. Diploma	5	4					4						
Ebhart, Amanda ICP Analyst AA Liberal Arts	2	2mo					2	2		2	2	2	
Foster, Desmond Organic Extractions Supv. H.S. Diploma	21	15			Also 5 years field & stack sampling *								
					2	5	19	7		6			
Freeman, Jennifer GC Analyst B.S. Micro Biology	4	3			2	2	2	2					
Gillund, Travis Laboratory Technician A.S. Chemistry	7.5	3mo			Also 1.5 years field sampling *								
							8		3	2			
Godbold, Linda GC/MS Analyst A.S. Medical Technology	12	8	8	5				3				3	
Harris, Robert Laboratory Director B.S. Microbiology	26		4	3	10		15	18	2		15		
Amanda Ham Inorganic Lab Technician H.S. Diploma	2	2					2	2		2	2	2	
Hayes, David RAD Chemistry Analyst B.S. Environmental Science	2	2					2	2					2
Hoke, Diana Organic Program Manager B.A. Chemistry	14		13	6	13		3	11		2			
Hoover, Chuck QA/QC Officer B.A. Biology/Chemistry					Also 10 yrs Project Management								
	22	11	2	2	5		8	7		5	4		

	Total Years Experience												
	Total Experience in Laboratory Field	Current Position	GC/MS	Purge & Trap	Gas Chromatography	High Performance Liquid Chromatography	Sample Preparation	Data Review	Inductively Coupled Plasma	Inorganic Analysis	Atomic Absorption	Data Reporting	Nuclear Chemistry
Julie Howard Inorganic Lab Technician B.S. Biology	2	2					2	2			2	2	
KariKari, Joseph GC Analyst B.S. Biochemistry	13	3	2	10	9		3	11		1	1	10	
Kidd, Gary ICP Analyst H.S. Diploma	13	13					6	2	3	7	7		
Klepperich, Debra Laboratory Technician High School	7	8mo											
Landtroop, Bryon GC Analyst High School Diploma	6	2		2	2		3	2					
Lynch, Ken Inorganic Wet Chemistry Analyst B.A. History/B.S. Chemistry	10	3						2		3			
Markham, Steve Laboratory Manager B.S. Zoology	22	2					13	13	10	13	3		
Martindale, Terry GC Chemist B.S. Chemistry	17	1	1	11	17	2	16	17	2	2	2		
Ralph, Curt GC/MS Analyst Trainee B.S. Environmental Science	2						2						
Rule, Jeff Dioxin Analyst B.S. Biochemistry	3	2	2		2		1	2					
Sein, Gideon Inorganic Analyst MB.ChB. Medicine M.S. Biological Science	6	2	3		2	2	3	2		1		1	
Self, Melissa Inorganic Lab Technician GED	1	1					1						
Shringarpure, Jayant Technical Director Ph.D. Organic Chemistry	20	7	18	18	18	5	5	20					
Spriggs, Jason Sample Receiving Supervisor H. S. Diploma	2	2											
Staggs, Randy Project Officer B.S. Biology	18	10			3		5	14		15	5		
Surface, Kert Nuclear Chemistry Manager B.S. Geology	12	4					12	12	1	3	1	9	9
Ta, David Analyst B.S. Biochemistry B.S. Chemical Engineering	2	2						2		2		2	
Turner, Susan Wet Chemistry Supervisor A.S. Chemistry	21	2	1		Also		1				15	15	5

	Total Years Experience												
	Total Experience in Laboratory Field	Current Position	GC/MS	Purge & Trap	Gas Chromatography	High Performance Liquid Chromatography	Sample Preparation	Data Review	Inductively Coupled Plasma	Inorganic Analysis	Atomic Absorption	Data Reporting	Nuclear Chemistry
					13	2	4	10					
Williamson, Janet VOA Supervisor M.S. Analytical Chemistry	19	3	8	6	11	3	6	6			2		

Appendix B

Logbook Standard Operating Procedures

Details of SWLO's logbook maintenance procedures are found in the following standard operating procedures:

Title	Document ID
Maintenance of Zone Logbooks	SWL-GA-128
Air Standard Traceability	SWL-OA-105
Volatile Standard Traceability	SWL-OV-109
Dioxin Standard Traceability	SWL-OD-109
Guide to Semivolatile GC/MS Standard Log	SWL-OS-309
Standards Receipt Traceability And Preparation	SWL-OP-202
Inorganic Traceability of Standards	SWL-IN-104
Air Logbook Maintenance	SWL-OA-104
Guide to Volatile GC/MS Run Log	SWL-OV-102
Dioxin Logbook Maintenance	SWL-OD-102
Semivolatile Logbook Maintenance	SWL-OS-305
Extraction Sheet Review	SWL-OE-104
Digestion/Distillation Log	SWL-IN-105

Appendix C-1

Ethics Policy Letter

Southwest Laboratory of Oklahoma, Inc.

Jack Wright
President

December 8, 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

Appendix C-2

Business Code of Conduct

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.

Appendix C-2 Cont.

Business Code of Conduct

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, DC 20460

September 1, 2001

[illegible]

Open letter to the environmental analytical laboratory community:

This letter is intended to draw your attention to issues of misconduct or unethical practices in analytical laboratories, related to inappropriate manipulation of laboratory data, that may also constitute laboratory fraud.

The EPA Office of Inspector General (EPA-OIG) has seen an increase in the number of allegations that environmental laboratories inappropriately manipulate laboratory data. This has been defined by my Office as: "The intentional falsification of analytical and quality assurance results." The falsification of environmental data undermines the ability of federal, state, tribal, and local governments, and the regulated community, to make environmental decisions necessary to protect the health and safety of the general public.

In a continuing effort to deter these practices, the EPA-OIG is working closely with EPA's Criminal Investigations Division, other federal investigative organizations, and the Department of Justice to investigate and, as appropriate, prosecute all allegations of laboratory fraud. These efforts to date have resulted in substantial fines, penalties, incarcerations, federal suspension and debarment actions, and laboratory closures.

Of particular concern is the fraudulent practice of using improper manual integrations by chromatographic analysts to falsify test results in initial calibrations, continuing calibrations, and surrogate recoveries, to make the control samples appear to meet quality control requirements. The routine practice of laboratory management spot checking a percentage of manual integrations performed in your laboratories would send a strong message to your laboratory staff that you are committed to ensuring that they follow proper laboratory procedures, identify improprieties, and correct them immediately. All too often, EPA-OIG's laboratory fraud investigations involve significant data manipulations conducted by multiple analysts using many analytical methods over several years. These investigations can raise questions about the reliability of all the data from a laboratory.

A laboratory's best protection against fraudulent activity is the effective implementation of strong, proactive, and independent ethical practices and Quality Systems. The Quality System should clearly establish and identify acceptable and unacceptable analytical practices. In addition, data authenticity checks should be incorporated into laboratory peer reviews, data verification, and/or Quality Assurance Officer's reviews to assure product integrity and to limit liability and exposure to "rogue analysts."

There are many factors that can contribute to misconduct: poor training, ineffective ethics programs, shrinking markets for analytical services, and greater economic incentives for laboratories to implement cost-cutting measures. Nevertheless, laboratory management is responsible for maintaining effective operations. To better ensure proper conduct, management can:

- Create a corporate environment conducive to quality operations, ethical standards, personal integrity, and accountability. This environment contributes to effective fraud prevention beyond anything industry standards and government oversight can accomplish.
- Plan for routine "down time" to perform instrument maintenance and repairs to meet method quality control requirements. These efforts can be marketed as a bulwark of quality assurance and technical ability.
- Acknowledge that improving quality promotes profits and an increase in business because a reputable laboratory *attracts* business.

I encourage you to evaluate your Quality System, with particular attention to the detection and prevention of improper laboratory activities. OIG staff are available to work with the environmental laboratory community and other stakeholders to encourage ethical laboratory operations and to prevent fraud. Any inquiries or comments regarding laboratory fraud should be directed to Earnest Dashiell, Acting Assistant Inspector General for Investigations, at (202) 260-3404.

Sincerely,

/s/

Nikki L. Tinsley

Appendix C-3

Business Code of Conduct Acknowledgement

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: _____

Form

Appendix C-4

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 date 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 contains 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 secrets of clients; client's data; and 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

Pace Analytical®
www.pacelabs.com

Pace Analytical Services, Inc
1700 West Albany
Broken Arrow, OK 74012
Phone: 918.251.2858
Fax: 918.251.2599

GA-101

The Standard Operating Procedures (SOPs) contained herein are adopted by Pace Analytical Services.

These SOPs will be followed until superceded by Pace Analytical Services SOPs.

Signature:	Signature:	Signature:
Chuck Hoover Quality Assurance Officer Pace Analytical Services, Inc. Broken Arrow, Oklahoma	Roger C. Splinter Dir. of Quality, Safety, Training Pace Analytical Services, Inc. Minneaplolis, MN 55414	Dave Neal General Manager Pace Analytical Services, Inc. Lenexa, KS

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

Standard Operating Procedure

Preparation, Review, Revision, and Control of Documents

Document No.: SWL-GA-101

Rev No. / Date: Rev. No.: 3.1 — 12/06/02

OFFICIAL COPY RED STAMP INDICATES ORIGINAL

APPROVALS	DATES
Procedure Prepared By	Date 12/06/02
Laboratory Manager or Director	Date 12/09/02
Laboratory Safety Officer	Date 12/06/02
Laboratory QA/QC Officer	Date 12/06/02

(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.: 2611 Issued to: TT NUS-PA Date: 10/17/03 QA: CAH

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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 compose each procedure.

2.2 Procedures which are administrative or non-analytical in nature, fall under the category "General" procedures and shall be reviewed and approved by the SOP author (person who prepared SOP), 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 SOP author, the responsible Laboratory Program Manager/Section Supervisor; Laboratory Safety Officer; and the QA/QC Officer.

2.3 It is the responsibility of the Document Control Officer to distribute controlled copies of new SOPs to the departments within the effective date on the cover of the SOP. This distribution will allow for training of the appropriate personnel who will implement the SOP. The Document Control Officer will then follow up with the department head to ensure that training documentation has been completed within the designated time frame.

2.4 It is the responsibility of the department head and/or supervisor to ensure that training and training documentation is completed within the time frame specified for implementation and to return the training documentation for new SOPs and/or major revisions to existing SOPs.

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

2.6 All re-training shall occur with each major SOP revision: i.e. 1.0, 2.0, etc. SOPs undergoing minor changes do not require re-training.

ocedures shall be reviewed annually. Procedures not requiring any
marked on the "*Request for New, Revised, or Retired Documentation*"
. A copy of this form documenting the review will be distributed
f the SOP.

xisting procedures are subject to the same preparation, review, and
as new original procedures.

ocuments requiring "Retired" status must be indicated on the "*Request
, or Retired Documentation*", and shall be routed to the DC Officer for
istribution of retired inserts.

ent shall assign the responsibilities of Document Control to a member of
staff, who must be proficient in the use of MS Word, MS Excel, and
er. The designated individual will be responsible for the controlled
maintenance of all documents as described herein.

2.7 All laborato
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with all cop

2.8 All revision
approval sy

2.9 All procedu
for New, Re
processing

2.10 The QA De
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distribution

3.0 EQUIPM

oftware; Velo-Binder; Document Stamps

MS Offic

NS

4.0 PRECAU

uments, which are not recovered: Reference, SWL-GA-105. *"Corrective Actions"*

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5.0 PROCEI

n of New SOPs

5.1 In

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

Th
Q
ba

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.

5.

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

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, and Retired SOP information.

5.

Note: For New SOPs, the Document # portion of the form shall be filled out by the DC Officer. 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 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 DC Officer, 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 DC Officer, the file can be e-mailed.

5.1.8 The DC Officer 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 DC Officer 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").

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 DC Officer 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.

5.1.9 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 DC Officer 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 DC Officer. 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 major changes are required, record as much information as possible on the form. If necessary, attach any additional instructions or related forms to this request.

NOTE: SOPs undergoing major revisions require that the relevant personnel undergo re-training. Major revisions are denoted as follows: 1.0, 2.0, 3.0, etc. Minor revisions are as follows: 1.1, 1.2, 2.1, etc.

5.2.2 The DC Officer will process all requests in order of priority as listed on the "Overdue Standard Operating Procedures" report, or as specified by Department Supervisor, or management.

5.2.3 Once completed the DC Officer 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 DC Officer 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 DC Officer 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 DC Officer 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 DC Officer must mark out the SOP Document ID with an orange highlighter, to indicate that the SOP was updated and is being circulated for signatures.

5.2.5 Once SOP is returned to the DC Officer 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 DC Officer 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 request form is then submitted to the DC Officer for processing.

5.3.1.2 The DC Officer 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 the other. 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 DC Officer will prepare them for distribution.

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

NOTE: For consistency, the yellow highlighter must be used.

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

5.4.3 The Officer 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. The letter 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.

5.4.7 A quarterly audit will be performed by the Document Control Officer or quality assurance Officer or Designee to ensure that the SOPs in the laboratory are the latest and current revisions. Discrepancies will be noted to the QAO and will be corrected.

5.5 Scheduled Review of Controlled Documents

5.5.1 The 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 reviewed and/or updated and given a new revision/review 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 corresponding 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 DC Officer 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 and the strictest QC are 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 SOP SWL-GA-114 , "*Hazardous Waste Management Plan*" should be included, along with any additional information not included in said 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 necessary 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

SWLO, INC. / AATS, INC.	GA-Series: SWL-GA-101
Preparation, Review, Revision, and Control of Documents	Rev. No.: 1.0 — 05/24/9

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)

❺ 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

NOTE: Use quotation marks and italics for books, SOPs, etc. For forms/logs, capitalize first letter of each word in title (no italics or quotations).

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.

Set Right tab at 7 inches.

Subsection line spacing at single. Bolded No.'s

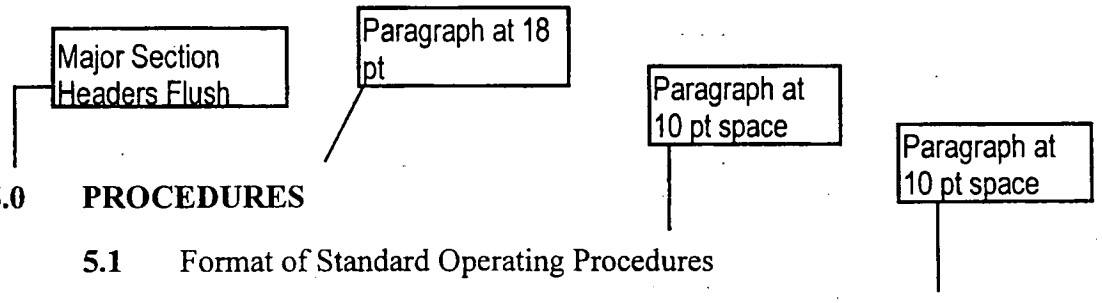
Pagination for TOC is i, ii, iii.

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



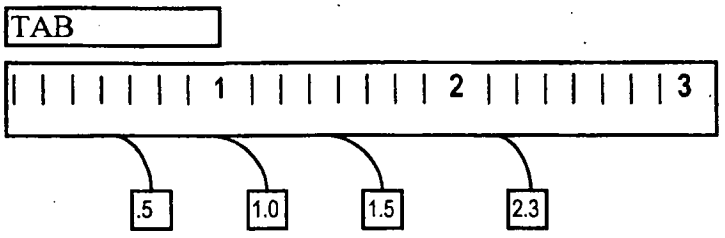
5.0 PROCEDURES

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.



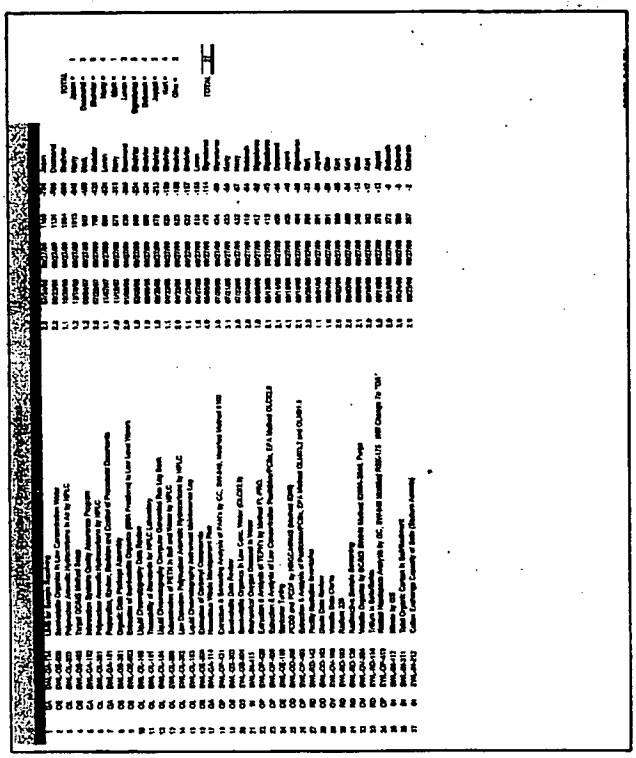
1st Level **1.0** → → →**0.5"** - SECTION TITLE Hanging Indents will be same as start of text

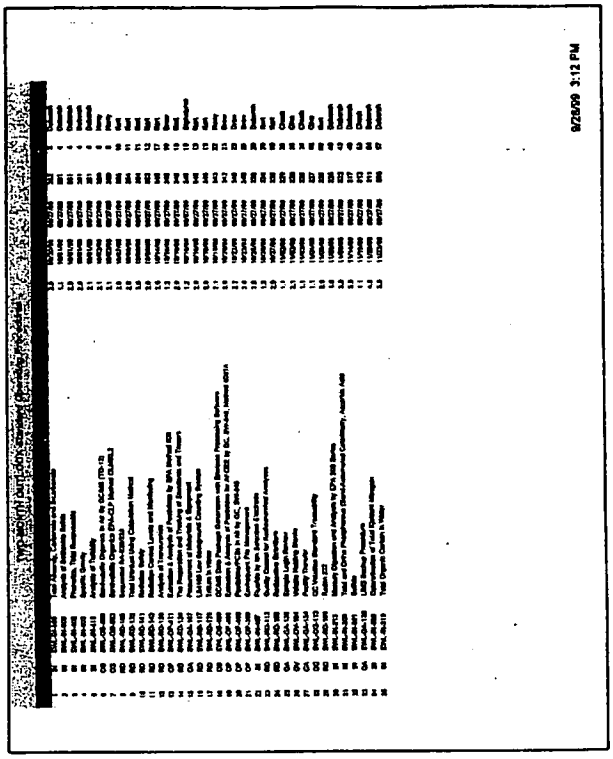
2nd Level No **1.1**→ →**0.5"** → →**1.0"** - Text - Hanging Indent at 1.0"

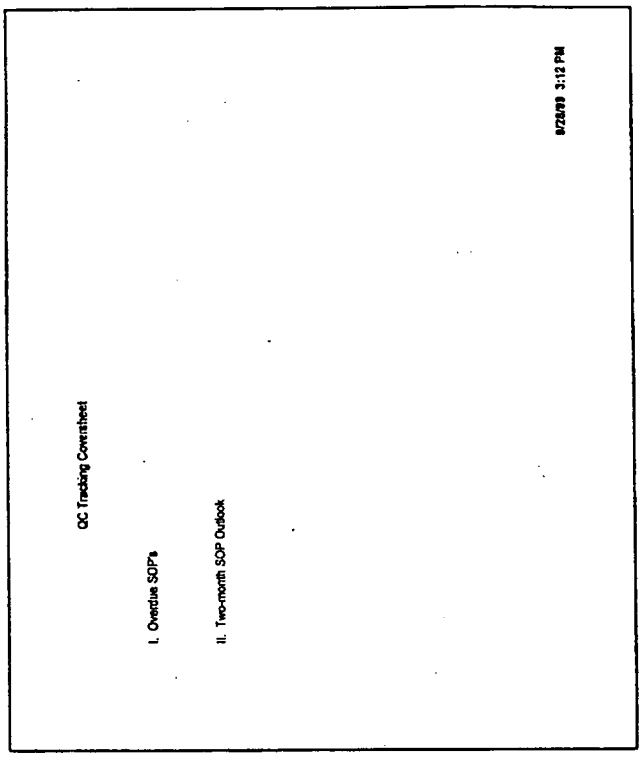
3rd Level No. **1.1.1**→ → → → **1.0"** → **1.5"** - Text - Hanging Indent at 1.5"

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).

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).

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).

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

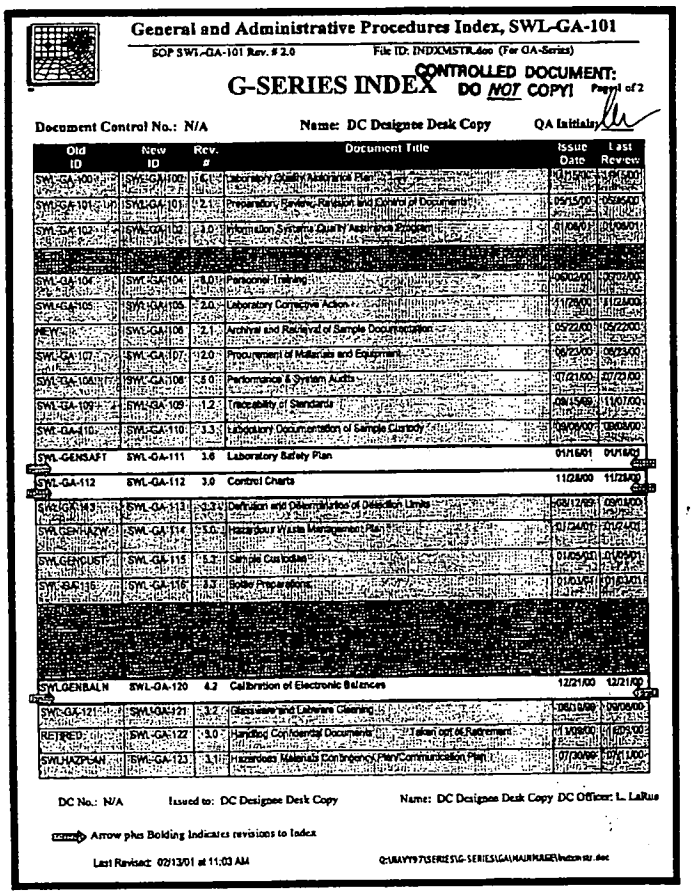
Date

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: INDXMSTR.doc (For GA-Series)

CONTROLLED DOCUMENT: DO NOT COPY!

G-SERIES INDEX Page 1 of 2

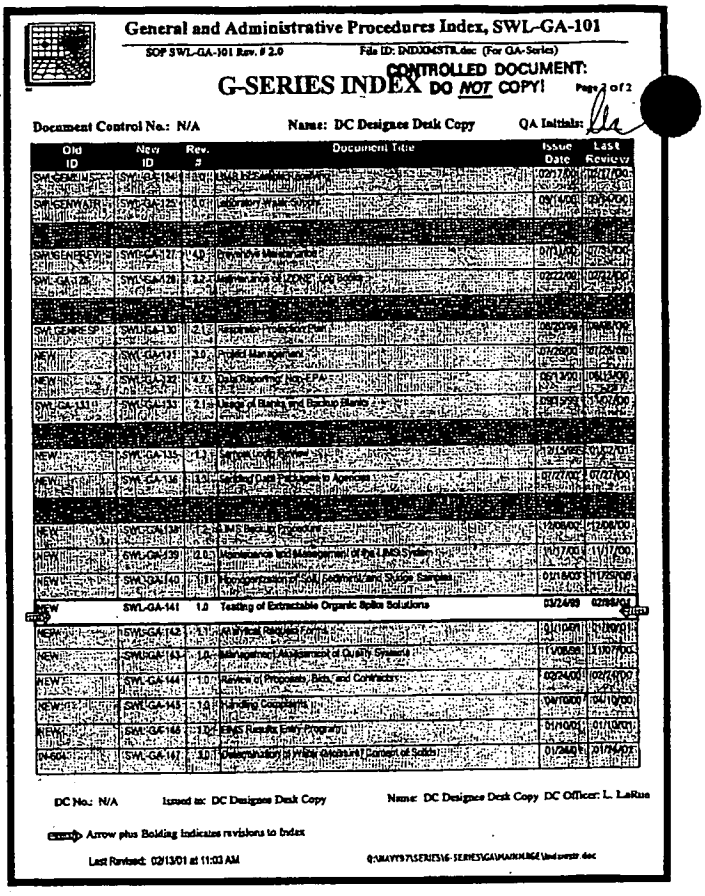
Document Control No.: N/A Name: DC Designee Desk Copy QA Initials:

Old ID	New ID	Rev. #	Document Title	Issue Date	Last Review
SWL-GA-100	SWL-GA-100	2.0	Laboratory Quality Assurance Plan	01/15/00	01/15/00
SWL-GA-101	SWL-GA-101	2.1	Preparation, Review, Revision and Control of Documents	05/15/00	05/15/00
SWL-GA-102	SWL-GA-102	3.0	Information Systems Quality Assurance Program	01/04/01	01/04/01
SWL-GA-104	SWL-GA-104	3.0	Personnel Training	06/02/00	06/02/00
SWL-GA-105	SWL-GA-105	2.0	Laboratory Corrective Action	11/29/00	11/29/00
NEW	SWL-GA-106	2.1	Archival and Retrieval of Sample Documentation	05/22/00	05/22/00
SWL-GA-107	SWL-GA-107	2.0	Procurement of Materials and Equipment	06/23/00	06/23/00
SWL-GA-108	SWL-GA-108	2.0	Performance & System Audits	07/21/00	07/21/00
SWL-GA-109	SWL-GA-109	1.2	Traceability of Standards	08/15/99	11/07/00
SWL-GA-110	SWL-GA-110	3.3	Laboratory Documentation of Sample Custody	10/09/00	10/09/00
SWL-GENSAFT	SWL-GA-111	3.0	Laboratory Safety Plan	01/16/01	01/16/01
SWL-GA-112	SWL-GA-112	3.0	Control Charts	11/28/00	11/28/00
SWL-GA-113	SWL-GA-113	3.2	Definition and Determination of Detection Limits	09/12/99	09/01/00
SWLGENHAZW	SWL-GA-114	3.0	Hazardous Waste Management Plan	01/24/01	01/24/01
SWLGENDUST	SWL-GA-115	3.3	Sample Custodian	01/05/01	01/05/01
SWL-GA-116	SWL-GA-116	3.3	Bottle Preparations	01/03/01	01/03/01
SWLGENBALN	SWL-GA-120	4.2	Calibration of Electronic Balances	12/21/00	12/21/00
SWL-GA-121	SWL-GA-121	3.2	Glassware and Labware Cleaning	09/10/99	09/09/00
RETIRED	SWL-GA-122	3.0	Handling Confidential Documents	11/09/00	11/09/00
SWLHAZPLAN	SWL-GA-123	3.1	Hazardous Materials Contingency Plan/Communication Plan	07/20/99	07/21/00

DC No.: N/A Issued to: DC Designee Desk Copy Name: DC Designee Desk Copy DC Officer: L. LaRue

Arrow plus Bolding Indicates revisions to Index

Last Revised: 02/13/01 at 11:03 AM Q:\MAY97\SERIES\G-SERIES\GA\MAINMAGE\Indexmstr.doc



General and Administrative Procedures Index, SWL-GA-101

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

CONTROLLED DOCUMENT: DO NOT COPY!

G-SERIES INDEX Page 2 of 2

Document Control No.: N/A Name: DC Designee Desk Copy QA Initials:

Old ID	New ID	Rev. #	Document Title	Issue Date	Last Review
SWLGENEMLIM	SWL-GA-124	3.0	LMS Hazardous Sampling	02/17/00	02/17/00
SWLGENWASR	SWL-GA-125	3.0	Laboratory Waste Disposal	09/14/00	09/06/00
SWLGENEQPM	SWL-GA-127	1.0	Equipment Maintenance	01/10/00	01/10/00
SWL-GA-128	SWL-GA-128	2.2	Maintenance of LDDNF Lab Bottle	02/22/00	02/22/00
SWLGENRESP	SWL-GA-130	2.1	Respirator Protection Plan	06/20/99	06/06/00
NEW	SWL-GA-131	3.0	Project Management	01/04/00	01/04/00
NEW	SWL-GA-132	4.0	Data Reporting: Non-EPA	06/13/00	06/13/00
SWL-GA-133	SWL-GA-133	2.1	Usage of Blanks and Backup Blanks	09/09/99	11/07/00
NEW	SWL-GA-135	1.3	Sample Logic Review	02/15/00	02/07/01
NEW	SWL-GA-136	1.3	Subcontract Data Packages to Agencies	02/27/00	02/27/00
NEW	SWL-GA-138	1.0	LMS Backup Procedure	12/06/00	12/06/00
NEW	SWL-GA-139	2.0	Maintenance and Management of the LMS System	10/07/00	11/17/00
NEW	SWL-GA-140	1.1	Reorganization of Soil Sediment/Land Sludge Samples	01/18/00	01/05/00
NEW	SWL-GA-141	1.0	Testing of Extractable Organic Spike Solutions	03/24/99	02/08/00
NEW	SWL-GA-142	1.1	GA Virtual Required Forms	01/10/01	01/10/01
NEW	SWL-GA-143	1.0	Management Assessment of Quality Systems	11/08/00	11/07/00
NEW	SWL-GA-144	1.0	Review of Proposals, Bids, and Contracts	02/24/00	02/07/01
NEW	SWL-GA-145	1.0	Handling Complaints	04/10/00	04/10/00
NEW	SWL-GA-146	1.0	LMS Results Entry Program	01/10/01	01/10/01
DN-60A	SWL-GA-147	3.0	Determination of Water Content of Solids	01/24/01	01/24/01

DC No.: N/A Issued to: DC Designee Desk Copy Name: DC Designee Desk Copy DC Officer: L. LaRue

Arrow plus Bolding Indicates revisions to Index

Last Revised: 02/13/01 at 11:03 AM Q:\MAY97\SERIES\G-SERIES\GA\MAINMAGE\Indexmstr.doc

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

MASTER DISTRIBUTION LIST - SOP

Standard Distribution Information for SWL Controlled Documents.

GA (General & Administrative) Procedural Documents

DC Dept. receives 1 (non-controlled blank copy) & maintains the Master Copies

Administrative Personnel	DC No.
Chuck Hoover	(DC-001)
Rob Harris	(DC-003)
Steve Markham	(DC-012)
Jan Travis	(DC-014)
Mark Smith	(DC-015)
Keith Sims	(DC-017)
Jason Spriggs	(DC-023)

DC023 Only receives 2-Controlled Copies of the following:
SWL-GA-100 SWL-GA-101 SWL-GA-105 SWL-GA-106 SWL-GA-110 SWL-GA-111
SWL-GA-114 SWL-GA-115 SWL-GA-116 SWL-GA-123 SWL-GA-124 SWL-GA-127
SWL-GA-128 SWL-GA-130 SWL-GA-134 SWL-GA-135

Randy Staggs	(DC-024)
Sandy Grovenstein	(DC-026)

DC-024 & DC-026 Both receive only 1-copy of the following:
SWL-GA-100 SWL-GA-116 SWL-GA-131 SWL-GA-135 SWL-GA-142

Barbara Forrester	(DC-027)
Pauline Lewis	(DC-028)

Pauline receives only 1-Copy of GA106 — No other SOPs

Technical Personnel	DC No.
Harry Borg	(DC-004)
Drew Cowan	(DC-005)
Jim Whitaker	(DC-006)
Jayant Shringarpure	(DC-007)
Deborah Inman	(DC-008)

Deborah receives all GA's including 4-copies of SWL-GA-100.

Kert Surface	(DC-009)

Kert receives all GA's plus 1-extra controlled copy of the following:
SWL-GA-100 SWL-GA-110 SWL-GA-114 SWL-GA-115

Desmond Foster	(DC-016)

Desmond receives all GA's plus 2-extra copies of SWL-GA-121.

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IN (Inorganics) Procedural Documents

DC Dept. receives 1 (non-controlled blank copy) & maintains the Master Copies

Personnel	DC No.
Chuck Hoover	(DC-001)
Steve Markham	(DC-012)
Barbara Forrester	(DC-027)
Deborah Inman	(DC-008)

See attached for Deborah's SOPs

Desmond Foster	(DC-016)

Desmond receives 2-copies of the following SOPs: SWL-IN-403 SWL-IN-409 SWL-IN-604

RD (Radiochemistry) Procedural Documents

DC Dept. receives 1 (non-controlled blank copy) & maintains the Master Copies

Personnel	DC No.
Chuck Hoover	(DC-001)
Steve Markham	(DC-012)
Barbara Forrester	(DC-027)
Kert Surface	(DC-009, DC-021, & DC-022)

Kert has 3-DC numbers as noted above. He should have 1-copy for each number; velo-bind the copy for DC-022.

OA (Organic Air) Procedural Documents

DC Dept. receives 1 (non-controlled blank copy) & maintains the Master Copies

Personnel	DC No.
Chuck Hoover	(DC-001)
Steve Markham	(DC-012)
Barbara Forrester	(DC-027)
Harry Borg	(DC-004)
Jayant Shringarpure	(DC-007)

Jayant gets 3-copies of all the OA SOPs.

OD (Organic Dioxin) Procedural Documents

DC Dept. receives 1 (non-controlled blank copy) & maintains the Master Copies

Personnel	DC No.
Chuck Hoover	(DC-001)
Steve Markham	(DC-012)
Barbara Forrester	(DC-027)
Harry Borg	(DC-004)
Jayant Shringarpure	(DC-007)

Jayant gets 2-copies of all the OD SOPs.

Desmond Foster	(DC-016)

Desmond receives 2-copies of the following:
SWL-OD-102 SWL-OD-202 SWL-OD-203 SWL-OD-204 SWL-OD-205 SWL-OD-206

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OE (Organic Extraction) Procedural Documents

DC Dept. receives 1 (non-controlled blank copy) & maintains the Master Copies

Personnel	DC No.
Chuck Hoover	(DC-001)
Steve Markham	(DC-012)
Barbara Forrester	(DC-027)
Harry Borg	(DC-004)
Desmond Foster	(DC-016)

Desmond receives 2-copies of all the OE SOPs.

OG (Organic Volatiles by GC) Procedural Documents

DC Dept. receives 1 (non-controlled blank copy) & maintains the Master Copies

Personnel	DC No.
Chuck Hoover	(DC-001)
Steve Markham	(DC-012)
Barbara Forrester	(DC-027)
Harry Borg	(DC-004)
Jim Whitaker	(DC-006)
Desmond Foster	(DC-016)

Desmond receives 2-copies of SWL-OG-106 only — No other SOPs.

OL (Organic HPLC) Procedural Documents

DC Dept. receives 1 (non-controlled blank copy) & maintains the Master Copies

Personnel	DC No.
Chuck Hoover	(DC-001)
Steve Markham	(DC-012)
Barbara Forrester	(DC-027)
Harry Borg	(DC-004)
Drew Cowan	(DC-005)
Desmond Foster	(DC-016)

Desmond receives 2-copies of the following:
SWL-OL-200 SWL-OL-201 SWL-OL-202 SWL-OL-203 SWL-OL-204 SWL-OL-206

OP (Organic Pesticides) Procedural Documents

DC Dept. receives 1 (non-controlled blank copy) & maintains the Master Copies

Personnel	DC No.
Chuck Hoover	(DC-001)
Steve Markham	(DC-012)
Barbara Forrester	(DC-027)
Harry Borg	(DC-004)
Drew Cowan	(DC-005)
Desmond Foster	(DC-016)

Desmond receives 2-copies of the following:
SWL-OP-301 (Will phase Desmond out of receiving the 400's Series for OP.)

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OS (Organic Semivolatile) Procedural Documents

DC Dept. receives 1 (non-controlled blank copy) & maintains the Master Copies

Personnel	DC No.
Chuck Hoover	(DC-001)
Steve Markham	(DC-012)
Barbara Forrester	(DC-027)
Harry Borg	(DC-004)
Desmond Foster	(DC-016)

Desmond receives 2-copies of the following:
SWL-OS-501 SWL-OS-506 SWL-OS-509

OV (Organic Volatile) Procedural Documents

DC Dept. receives 1 (non-controlled blank copy) & maintains the Master Copies

Personnel	DC No.
Chuck Hoover	(DC-001)
Steve Markham	(DC-012)
Barbara Forrester	(DC-027)
Harry Borg	(DC-004)
Jim Whitaker	(DC-006)

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

Typist: LLR

ATTACHMENT 1

IN-SERIES DOCUMENT CONTROLLED COPIES

Example

2 Master Sets

PLUS

4 additional copies of SWL-IN-100

TOTAL: 6

No.	Doc. I.D.	Document Title
4	SWL-IN-100	Calibration of Eppendorfs
2	SWL-IN-102	Inorganic Data Package Assembly
4	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 Analysis
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 Analysis
4	SWL-IN-205	ICP SW3010A / 3050B Digestion and SW6010B Analysis
2	SWL-IN-207	Mercury, SW846-7470A / 7471A, Digestion and Analysis
2	SWL-IN-210	GFAA, SW846-7000 Series Analysis
2	SWL-IN-212	Cation Exchange Capacity of Soils (Sodium Acetate)
4	SWL-IN-213	ICP 200 Series Digestion and Analysis
1	SWL-IN-215	Mercury Digestion and Analysis by EPA 200 Series
3	SWL-IN-217	GFAA Digestion and Analysis 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	Analysis 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-313	Cyanide by SM4500-CN-1
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
1	SWL-IN-405	pH Electrometric Measurement (Test Codes IN200 & HW200)
1	SWL-IN-406	Determination of Colorimetric Hexavalent Chromium (MT169)

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No.	Doc. I.D.	Document Title
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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NOTE: Actual size is 8 ½ x 11 inches. (Standard letter size).

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

Standard Operating Procedure

Preparation, Review, Revision, and Control of Documents

Document No.: SWL-GA-101

Rev No. / Date: Rev. No.: 3.1 — 12/05/02

APPROVALS	DATES
Procedure Prepared By	Date
Laboratory Manager or Director	Date
Laboratory Safety Officer	Date
Laboratory QA/QC Officer	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.:_____ Issued to:_____ Date:_____ QA:_____

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

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. 4.0 10/31/02 FILE ID: GA101A2a.doc

[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]

Page 1 of 1

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).

Laboratory Master Logbook Log, SWL-GA-101

GENERAL ADMINISTRATIVE

SOP SWL-GA-101, REV. 4.0 10/31/02 FILE ID: GA101A2b.doc

Logbook Number						
Inst. Initials		Logbook Title	Start Date	End Date	Entry	Last Page

Key:

RL = Run Log
EL = Extractions Log
CC = Calibration Checks
SL = Standards Log
PL = Prep Log
MSPL = Multi-standard Preparation Log
PMS = Preventative Maintenance Schedule

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).

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**

TSS/TDS Logbook, Ref: SWL-IN-600

INORGANICS DEPARTMENT

TSS/TDS Logbook (IN600A1.doc) SOP SWL-IN-600, Rev: 5.0 07/11/01
Method References: EPA 160.1/160.2/160.3, SM 2540B/2540C/2540D

Book # IN-600-TSSTDSLog-__

Test Code: _____ Date of Analysis: _____ Time: _____ Batch ID: _____
Date/Time In: _____ Date/Time Out: _____ Temp. In: _____ Temp. Out: _____ Analyst's Initials: ____

Dish or Crucible #	Sample ID	Comments	B Dish or Crucible Weight	C Sample Volume	A Dish or Crucible + Residue 1st	A Dish or Crucible + Residue Final	D Dish or Crucible + Residue 1st	D Dish or Crucible + Residue Final	A-B A-D D-B	Dilution	Final Results	Units	% Rec % RPD

TDS: 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

TSS: Total Suspended Solids, mg/L = $\frac{(A-B) \times 1000}{C}$
Where: A = Weight of crucible dish + residue in mg.
B = Weight of crucible dish in mg
C = mL of sample used

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).

Typist: LLR

FLUORIDE STANDARD BATCH FORM

INORGANICS DEPARTMENT

FLUORIDE STD. BATCH FORM (IN414F1.DOC) SWL-IN-414, 4.0 12/21/01 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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NOTE: Actual size is 8 ½ x 11 inches. (Standard letter size).

INORGANICS DEPARTMENT

Analysis of Fluoride (SPADNS – IN130) Logbook, Ref: SWL-IN-414

Analysis of Fluoride (SPADNS – IN130) Logbook (IN414A2.doc) SOP SWL-IN-414, Rev: 4.0 12/21/01 Book # IN-414-SPADNS-____

Test Code: _____ Date of Analysis: _____ Time: _____ Batch ID: _____ Analyst's Initials: _____

Sample ID	T. V.	Matrix	Initial Wt. Vol.	Final Wt. Vol.	Dilution	Spike/Comments	Absorbance	Results	Units	% Rec.

Multiply readings by any dilution factor mode to obtain final results.

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

Typist: LLR

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GA-102

The Standard Operating Procedures (SOPs) contained herein are adopted by Pace Analytical Services.

These SOPs will be followed until superceded by Pace Analytical Services SOPs.

Signature:

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Quality Assurance Officer
Pace Analytical Services, Inc.
Broken Arrow, Oklahoma

Signature:

Roger C. Splinter
Dir. of Quality, Safety, Training
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General Manager
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Lenexa, KS

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

TM

Standard Operating Procedure

Information Systems Quality Assurance Program

Document No.: SWL-GA-102

Rev No. / Date: Rev. 5.0 — 03/14/03

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APPROVALS	DATES
Mark A. Sattler Procedure Prepared By	3-14-03 Date
Robert W. Harbu Laboratory Manager or Director	3/25/03 Date
Chuck Jones Laboratory Safety Officer	3/27/03 Date
Chuck Jones Laboratory QA/QC Officer	3/27/03 Date

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

Document Status

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

[✓] Official Copy OC No.: 2615 Issued to: TT NUS - PA Date: 10/17/03 Initials: CFH

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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 Access 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. This database is located in Access.
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, HPLC, and high Resolution GC/MS 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. It is reviewed, verified and approved. QC reports are generated and the data is downloaded to the SQL server 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 Organic CLP diskettes, which are generated from an in-house developed program – GC and GC/MS.
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 two 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.

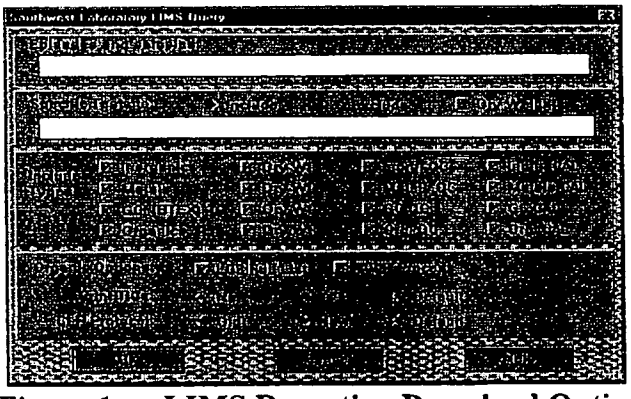


Figure 1 — LIMS Reporting Download Options

This screen is used to query or download specific Prep QC information. From the options 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

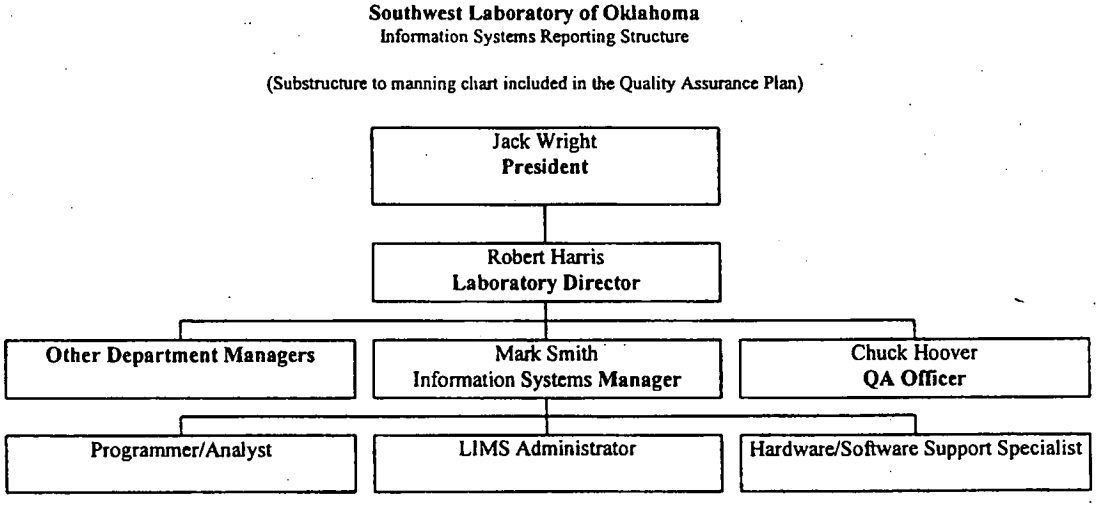
Data Manipulations

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. Refer to SWL-GA-146, "LIMS Results Entry Program", on how hand entry is performed.

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 3.

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

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

Upon system failure, the Hardware/Software Support Specialist should be notified. If after business hours or on weekends, contact them at home or through their pager. If the Hardware/Software Support Specialist is unavailable, contact the Information Systems Manager. If the Information Systems Manager is unavailable, contact the Laboratory Director.

SWLO maintains a backup NT server. This server may be used to replace any of the NT servers should a catastrophic failure occur. The necessary data and programs will be installed from the most recent backup of the system.

The Hardware/Software Support Specialist takes the most recent full backup of the NT servers off site each day. If the Hardware/Software Support Specialist is absent, the Information Systems Manager will perform this task.

Equipment Hardware

All PCs are 386, 486 and Pentium Intel based processors and are located throughout the lab. The network type is and Ethernet 10BaseT using TCP/IP protocol.

SWLO has four NT servers as part of its LIMS system. These systems are described below.

SWLO1
- AMD 1.3GHz Processor
- 256 MB RAM
- (4) 60GB hard drives in RAID 0+1 configuration
- Exabyte Mammoth 2 Tape Drive

Usage: Main file server, primary domain controller, backup server.

SWLO2
Dell 1300
(2) PIII 750 MHz Processor
512 MB RAM
(4) 9GB hard drives in RAID 5 configuration

Usage: External IIS (Web server), Exchange server, backup domain controller, proxy server, VPN server

SWLO3
Dell 4500
(2) PIII 450 MHz Processor
512 MB RAM
(5) 18GB hard drives in RAID 5 configuration

Usage: File server, DHCP server, WINS server, Print server

SWLO4
AMD 900 MHz Processor
512 MB RAM
(4) 30GB hard drives in RAID 0+1 configuration

Usage: Internal IIS (WEB server), SQL server, Program server

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 (Attachment 5.) Hardware maintenance logs are maintained by the Hardware/Software Support Specialist for the main LIMS, Databases, and file servers. The logs are kept in spiral notebooks in the Server Room.

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, upgrade, or modification to computer systems that affect the laboratory reporting data is performed, all outdated hardware is removed from service. If the new hardware is not compatible with the replaced hardware, the replaced hardware 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.

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

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.

In House Software & Spreadsheet

Software Programming Request Forms

Completion of the Software Programming Request Form

A request for program development or modification is communicated through use of the Software Programming Request Form (Attachment 6). Outlined below are the fields of the form, and a description of the Software Programming Request procedure.

Name: Name of requesting individual

Date: Current date

Department: Department in which the requesting Individual is assigned

Date Needed By: The date by which the request is needed by. NOTE: ASAP will be used to request that high priority be given in scheduling, but does not guarantee immediate attention. If you have a request that requires immediate attention, submit the request to the Laboratory Director or Operations Manager for their approval.

Authorization by Dept. Mgr.: If the request is completed by hand, the department manager or their designee in case of absence, must sign the request. If the request is e-mailed, the requesting individual should get the approval of the department manager before the e-mail is sent. A carbon copy of and e-mailed request must be sent to the department manager.

Authorization by Administration: Laboratory Director or Operations Manager are to approve all new programs and major enhancements.

Type of request: Select one of the following: New Program, Enhancement, or Program Repair.

Explanation of Request: Give a description of the request. Multiple requests on a single form should be avoided unless the items are directly related.

Explanation of Verification: After the programming has been completed. The form will be returned to the requesting individual for verification. Give a description of how the request was verified.

Software Control Number, Program, Programmed by, and Date: To be completed by the IS dept.

Verified by: Signature of the individual who verified the request.

Pass and Date: Check the box if the verification passed. Enter the date the verification was complete.

Validated by and Date: To be completed by the QC dept.

Software Programming Request Procedure

1) Complete the Software programming request form through the Explanation of Request section. Obtain necessary authorization.
2) Submit the request along with supporting documentation to the Information Systems Manager. Supporting documentation includes forms or reports to be corrected or modified, screen dumps, program error messages, etc.
3) After the programmer has completed the request, the Software Programming Request Form will be returned to the requesting individual or department for verification.
4) The requesting individual or department will test the program to verify the objective of the request was met. If calculations were added or changed, the verifier must compare the results against results determined by another method. Complete the Explanation of Verification and Verified by sections of the form.
5) If the program fails verification, return the request to the programmer with an explanation of why the program failed. The cycle of programming/verification will continue until the program passes verification.
6) If verification passes, submit the completed form along with the verification documentation to the QC department for validation.
7) The QC department will review the verification process and results and sign the form approving the program for distribution.
8) The completed form and documentation is given to the LIMS Administrator for logging in the Software Control Logbook and filing.
9) The Software Control Logbook is an Excel Spreadsheet stored in SWLO1:\Robyn\Misc\Index.xls (see Attachment 7 for example).

New Program/Spreadsheet Development

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.

Training

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

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 6). 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 6.

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. The Validator will review the explanation of verification and the documentation provided by the data verifier. The Validator completes the "Validated by and date" sections of 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, person testing, and name of programmer.

All major software systems must have user documentation. User documentation may consist of manuals, SOPs, program "Help" screens, or Intranet pages. Intranet pages may be found through the "Departments," "Software Documentation" options.

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.

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 LIMS data modification 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 8) 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 9) 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, direction 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.

Historical software changes that should be archived are those changes that affect the calculation and reporting of data. For programs that are not checked into the PVCS Version Manager system, these historical software files are stored in the Progdev directory of the Computer share on the SWLO3 server. The Progdev directory contains subdirectories for each computer project. If the software product is checked into the PVCS version Manager System, then all historical versions of the software are maintained by the system.

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, through the Results Entry Program. Usage of the Results Entry Program is found in SOP SWLO-GA-146. 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 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 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.

Southwest Laboratory of Oklahoma also uses Web-Shield-X for Internet virus protection.

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.

Back-up Frequency

Automated backups are made daily for each Windows NT server. Tapes are verified for completeness as outlined in SWL-GA-139.

Data from acquisition systems is transferred to the main file server (SWLO1). The data backup will be performed the next scheduled backup after the data has been transferred to the file server. In the unlikely event that the main file server should go down, or is unavailable for more than 24 hours, data will be rerouted and backed-up using a secondary server. If transfer of data to the secondary server is not available, data backups will be performed on the acquisition systems.

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.

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

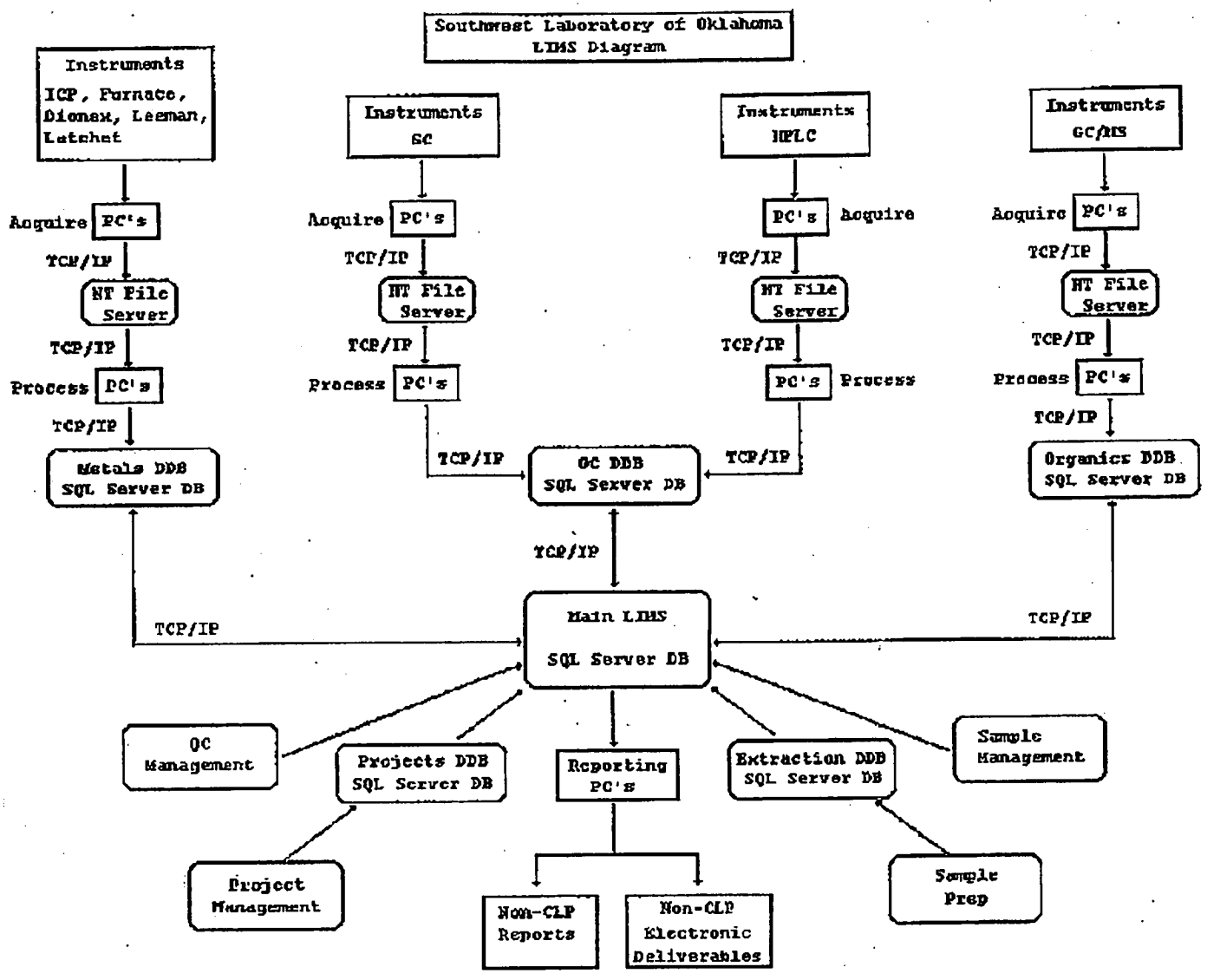
The IS Manager is responsible for the resolution of all customer complaints and corrective actions involving the computer department.

Records

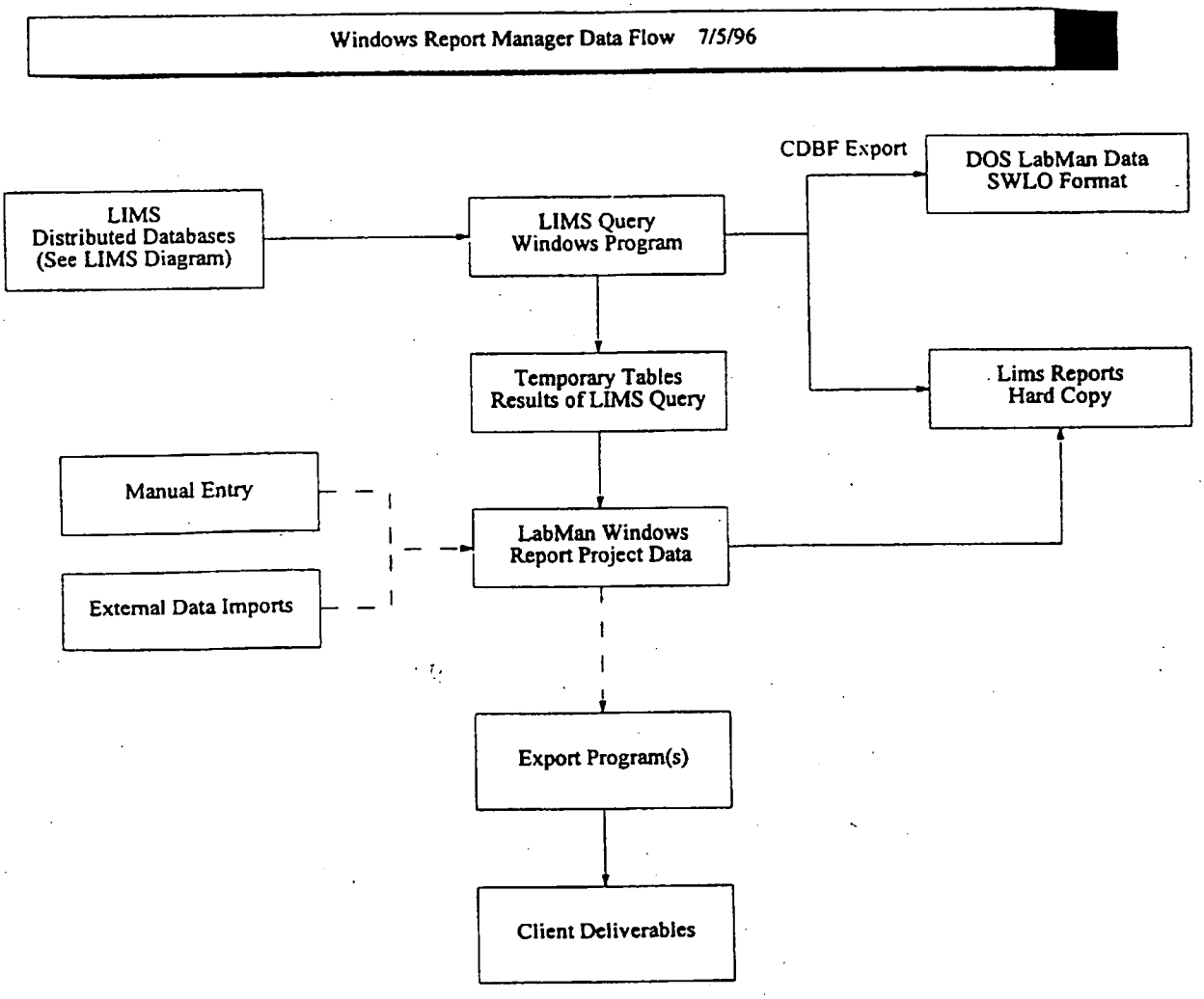
All records associated with the complaint or corrective action must be maintained as outlined in the complaints and corrective action procedures in the Laboratory Quality Assurance Program.

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:

- Information Systems 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.

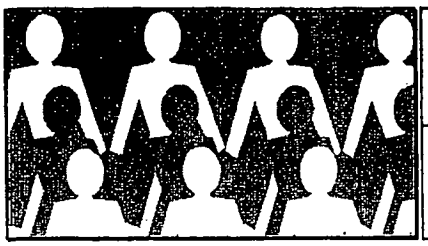
SUPERVISION RECEIVED
Work is occasional reviewed in progress, but is normally reviewed after-the-fact. Guided by established practiced and procedures but exercises independent judgement to achieve predetermined end results.

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.

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.

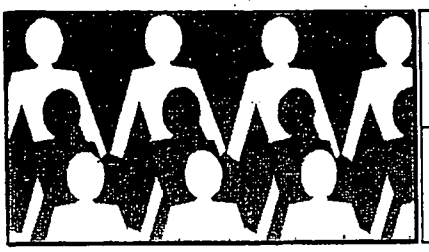
MAJOR DUTIES AND RESPONSIBILITIES
- Perform all system backup and archive procedures and check results. Review error reports and messages, and resolve problem situations.
- Facilitate data transfers to and from affiliated laboratories.
- Provide technical support for the LIMS systems. Maintain access 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

ACCOUNTING DEPARTMENT

EQUIPMENT LOCATION TRANSFER DOCUMENTATION

TM

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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[AC012-0492-01]

ATTACHMENT 5
Computer Service Request Form

COMPUTER SERVICE REQUEST

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 6
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:

ATTACHMENT 7
Software Control Logbook Form

IN100/07-09-97	filter.exe	
IN101/09-11-97	loadddb.exe	
IN102/09-11-97	qcform.exe	
IN102*	qcform.exe	
IN103/07-11-97	QCFORM.WB1:	ILCSW
IN104/07-11-97	QCFORM.WB1:	ILCSS
IN105/07-11-97	QCFORM.WB1:	IMSDW
IN106/07-11-97	QCFORM.WB1:	IMSDS
IN107/07-11-97	QCFORM.WB1:	IDPU
IN108/07-11-97	QCFORM.WB1:	FLCSW
IN109/07-11-97	QCFORM.WB1:	FLCSS
IN110/07-11-97	QCFORM.WB1:	FMSDW
IN111/07-11-97	QCFORM.WB1:	FMSDS
IN112/07-11-97	QCFORM.WB1:	FDUP
IN113/07-11-97	QCFORM.WB1:	TLCSW
IN114/07-11-97	QCFORM.WB1:	TMSDW
IN115/07-11-97	QCFORM.WB1:	ALCSW
IN116/07-11-97	QCFORM.WB1:	ALCSSS
IN117/07-11-97	QCFORM.WB1:	AMSDW
IN118-07-11-97	QCFORM.WB1:	AMSDS
IN119/07-11-97	QCFORM.WB1:	ADUP
IN120/07-11-97	QCFORM.WB1:	GWCOND\PH
IN121/07-11-97	QCFORM.WB1:	GSCOND\PH
IN122/07-11-97	QCFORM.WB1:	GLCSW
IN123/07-11-97	QCFORM.WB1:	GLCSS
IN124/07-11-97	QCFORM.WB1:	GMSDW
IN125/07-11-97	QCFORM.WB1:	GMSDS
IN126/07-11-97	QCFORM.WB1:	GDUPW
IN127/07-11-97	QCFORM.WB1:	GDUPS
IN128/07-11-97	QCFORM.WB1:	TRACELCW
IN129/07-11-97	QCFORM.WB1:	TRACELCS
IN130/07-11-97	QCFORM.WB1:	TRACEMSDW
IN131/07-11-97	QCFORM.WB1:	TRACEMSDS
IN132/07-11-97	QCFORM.WB1:	TRACEDUP
IN133*	AAIDLS.WB1	
IN134/02-04-98	ICPMDL.WB2:	TJAET1
IN135/02-04-98	ICPMDL.WB2:	TJAET2
IN136/02-04-98	ICPMDL.WB2:	TJA61E
IN137/02-04-98	CEC.WB1	
IN138*	IN090.WB2	
IN139/02-04-98	SULFIDE.WB1:	SULFIDE-WATERS
IN140/02-04-98	SULFIDE.WB1:	SULFIDE-SOILS
IN141/02-04-98	SULFIDE.WB1:	REACTIVE-WATERS
IN142/02-04-97	SULFIDE.WB1:	REACTIVE-SOILS
IN143/06-24-98	HARDCALC.WB1	
IN144/02-04-98	DETLIM.WB1:	PE5100IDL
IN145/02-04-98	DETLIM.WB1:	PE5100MDLS
IN146/02-04-98	DETLIM.WB1:	VARIANAIDLS
IN147/02-04-98	DETLIM.WB1:	VAR400MDLS

ATTACHMENT 8
Test Code Addition or Modification Request Form

Test Code Addition or Modification Request Form

COMPUTER SERVICE DEPARTMENT

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 9
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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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. 8.0 — 09/05/03

OFFICIAL COPY RED STAMP INDICATES ORIGINAL

APPROVALS	DATES
Melissa Crenick Procedure Prepared By	9-12-03 Date
Robert W. Harris Laboratory Manager or Director	9/12/03 Date
Chuck Jones Laboratory Safety Officer	9/5/03 Date
Chuck Jones Laboratory QA/QC Officer	9/5/03 Date

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

Document Status

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

[✓] Official Copy OC No.: 2611 Issued to: IT NUS-PA Date: 10/17/03 Initials: CAH

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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 Quality Assurance and Safety Training 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 6).

5.1.2.3 Initial proficiency certification and recertification shall be documented on a Proficiency Summary Form (Attachment 7) and form GA104A3 "Demonstration of Capability," (Attachment 8).

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). Included in this general training is ethics briefings, after which employees are asked to sign a "Code of Business Conduct" (Attachment 2) and a "Confidentiality Agreement" (Attachment 3).

5.2.2 Supervisors of new employees and those transferred from a different job function are required to fill out a "New Employee Training Plan" GA104A7 (see Attachment 5). The training plan 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 6) 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 Summary and Statement (see Attachments 7 and 8).

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 Laboratory Control 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 LCS 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. For full list spikes (e.g. 8260 / 8270) where 10% of the compounds are allowed out for LCS the same criteria applies for the IDC.

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 and Ethics. 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.

5.7 Training File Audits

5.7.1 Quarterly Training File Audits will be performed to ensure that employee training records are being maintained (See SWL-GA-108, Performance and System Audits for more details).

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 "Code of Business Conduct" adherence agreement shall be retained in the personnel records section of the QA file as a permanent record (see Attachment 2).

6.3 "Confidential Information Agreement" shall be retained in the personnel records section of the QA file as a permanent record (see Attachment 3).

6.4 "New Employee Training Plan" (see Attachment 5).

6.5 Form GA104A2 "Document Review and Understanding" will be retained in the personnel records section of the QA file as a permanent record (see Attachment 6).

6.6 Initial Demonstration of Accuracy and Precision (IDAP — see Attachment 7).

6.7 Form GA104A3 "IDC Certification Statement" 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.

IDC:.....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

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

Attachment 2: Code of Business Conduct.

Attachment 3: Confidential Information Agreement.

Attachment 4: Sample of Laboratory Job Description.

Attachment 5: New Employee Training Plan.

Attachment 6: Document Review and Understanding.

Attachment 7: Initial Demonstration of Accuracy and Precision (IDAP)

Attachment 8: Certification Statement (IDC)

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 Rev. # 8.0 09/05/03

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

Code of Business Conduct

Southwest Laboratory of Oklahoma, Inc.

Jack Wright
President

December 8, 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.

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, DC 20460

September 5, 2001

THE INSPECTOR GENERAL

Open letter to the environmental analytical laboratory community:

This letter is intended to draw your attention to issues of misconduct or unethical practices in analytical laboratories, related to inappropriate manipulation of laboratory data, that may also constitute laboratory fraud.

The EPA Office of Inspector General (EPA-OIG) has seen an increase in the number of allegations that environmental laboratories inappropriately manipulate laboratory data. This has been defined by my Office as: "The intentional falsification of analytical and quality assurance results." The falsification of environmental data undermines the ability of federal, state, tribal, and local governments, and the regulated community, to make environmental decisions necessary to protect the health and safety of the general public.

In a continuing effort to deter these practices, the EPA-OIG is working closely with EPA's Criminal Investigations Division, other federal investigative organizations, and the Department of Justice to investigate and, as appropriate, prosecute all allegations of laboratory fraud. These efforts to date have resulted in substantial fines, penalties, incarcerations, federal suspension and debarment actions, and laboratory closures.

Of particular concern is the fraudulent practice of using improper manual integrations by chromatographic analysts to falsify test results in initial calibrations, continuing calibrations, and surrogate recoveries, to make the control samples appear to meet quality control requirements. The routine practice of laboratory management spot checking a percentage of manual integrations performed in your laboratories would send a strong message to your laboratory staff that you are committed to ensuring that they follow proper laboratory procedures, identify improprieties, and correct them immediately. All too often, EPA-OIG's laboratory fraud investigations involve significant data manipulations conducted by multiple analysts using many analytical methods over several years. These investigations can raise questions about the reliability of all the data from a laboratory.

A laboratory's best protection against fraudulent activity is the effective implementation of strong, proactive, and independent ethical practices and Quality Systems. The Quality System should clearly establish and identify acceptable and unacceptable analytical practices. In addition,

data authenticity checks should be incorporated into laboratory peer reviews, data verification, and/or Quality Assurance Officer's reviews to assure product integrity and to limit liability and exposure to "rogue analysts."

There are many factors that can contribute to misconduct: poor training, ineffective ethics programs, shrinking markets for analytical services, and greater economic incentives for laboratories to implement cost-cutting measures. Nevertheless, laboratory management is responsible for maintaining effective operations. To better ensure proper conduct, management can:

- Create a corporate environment conducive to quality operations, ethical standards, personal integrity, and accountability. This environment contributes to effective fraud prevention beyond anything industry standards and government oversight can accomplish.

- Plan for routine "down-time" to perform instrument maintenance and repairs to meet method quality control requirements. These efforts can be marketed as a hallmark of quality assurance and technical ability.

- Acknowledge that improving quality promotes profits and an *increase* in business because a reputable laboratory *attracts* business.

I encourage you to evaluate your Quality System, with particular attention to the detection and prevention of improper laboratory activities. OIG staff are available to work with the environmental laboratory community and other stakeholders to encourage ethical laboratory operations and to prevent fraud. Any inquiries or comments regarding laboratory fraud should be directed to Emmett Dashiell, Acting Assistant Inspector General for Investigations, at (202) 260-3404.

Sincerely,

/s/

Nikki L. Tinsley

Printer-Friendly Version
Email this Story

Labs increasingly caught faking environmental tests

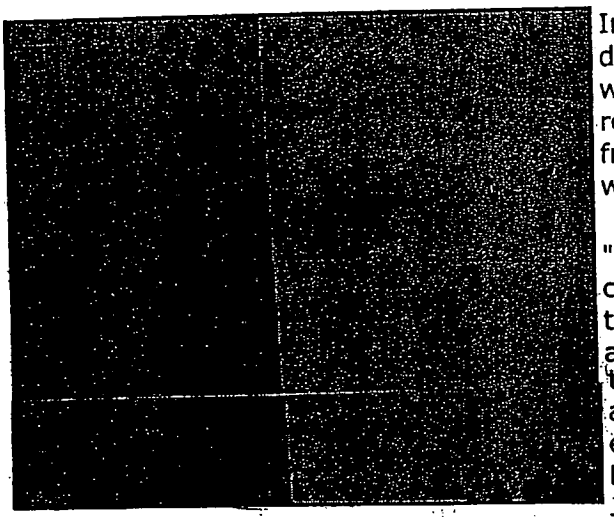
Jan 22, 2003

BY LARRY MARGASAK
THE ASSOCIATED PRESS

WASHINGTON - Private laboratories are increasingly being caught falsifying test results for water supplies, petroleum products, underground tanks and soil.

This hampers the government's ability to ensure Americans are protected by environmental laws, investigators say.

The fraud has caused millions of people to fill their cars with substandard gasoline that may have violated clean-air standards, or to drink water not properly tested for safety, the officials told The Associated Press.



In addition, officials making decisions at hazardous-waste cleanup sites have relied on companies that fraudulently tested air, water and soil samples.

"In recent years, what has come to our attention is that [nongovernment] labs are oftentimes in bed with the people who hired them, and conspired to commit environmental crime," said David Uhlmann, chief of the Justice Department's environmental crimes section.

The EPA's watchdog against fraud, Inspector General Nikki Tinsley, said the rise of lab fraud a disturbing trend.

"If it was my drinking water I'd consider it very serious," she said, declining to identify locations affected by the ongoing investigation.

Private laboratories test products that are regulated by anti-pollution laws, and the results allow companies to certify that they're meeting the requirements of environmental protection laws.

In one instance three years ago, investigators discovered fraudulent test results by contract employees at the Environmental Protection Agency's lab in Chicago. The head of the laboratory was transferred and the contractor, Lockheed Martin, was suspended from performing tests.

The Justice Department and Environmental Protection Agency have prosecuted dozens of employees and laboratories the past several years for fraudulent testing. Uhlmann, the Justice Department official, said the prosecutions have grown but statistics are not kept on lab-fraud cases.

The growing number of cases stretch from New England, where a chemist for municipal water made up test results, to Texas, where the government recently prosecuted the largest tester of underground fuel tanks.

Officials said they aren't certain whether an increasing number of labs are falsifying tests, or whether more are simply being caught.

Tinsley said there were numerous reasons for lab misconduct: poor training, ineffective ethics programs, shrinking markets and efforts to cut costs.

In some cases, the labs duped the companies that submitted samples for testing. In other instances, the companies were part of a conspiracy with the labs, officials said.

Sometimes the fraud includes "driveway tests," so-named because employees generate them in their own driveways, without visiting the facilities.

Whatever the case, lab fraud hampers an environmental protection system that frequently relies on voluntary compliance by companies backed by test results, officials said. Faked results can mislead regulators and the public into thinking they are being protected by laws when in fact companies are not abiding by the safeguards.

"If we can't rely upon science with supporting lab results, then we don't know what's out there for the public to eat or drink or use," said J.P. Suarez, the EPA's assistant administrator for enforcement and compliance assurance.

"When people may not be getting harmed, they may be getting ripped off, using products that are not what they're paying for. And companies are paying for services they're not getting," he said.

A recent example of fraudulent testing: Former environmental contractor James Edward Adams of Inman, S.C., was sentenced to 27 months in prison. His company, which provided testing services for underground storage tanks, directed employees to provide false test reports to owners and operators of petroleum tank facilities in South Carolina, North Carolina, Florida, Georgia, Virginia and Tennessee, prosecutors said.

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 3

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 4

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 5

New Employee Training Plan

NEW EMPLOYEE TRAINING PLAN

SWL-GA-104 REV.8.0 09/05/03 GA104A5

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 6

Document Review and Understanding

DOCUMENT REVIEW AND UNDERSTANDING,

SWL-GA-104

Personnel Training Form (GA104A6) SOP Rev. # 8.0 09/05/03

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 7

Initial Demonstration of Accuracy and Precision (IDAP)

Initial Demonstration of Accuracy and Precision

SWL-GA-104 7.0 08/25/01 Attachment 7 I:qcforms:\qc\prof\proficfm.wb2

Method:
Analyst:
Date:
Instrument:
Analytical Column:

Sample Prep Method:
Sample Prep Analyst:
Matrix: **Date Prep:**
Sample Weight or Volume:
Spike Weight or Volume:
Spike Conc. & Amount:

CONTAMINANT	TEST CONC.	QC 1	QC 2	QC 3	QC 4	X	ACCEPTANCE CRITERIA	RSD	ACCEPTANCE CRITERIA	PASS or FAIL
								ERR	<20% RSD	
								ERR	<20% RSD	
						ERR		ERR	<20% RSD	
						ERR		ERR	<20% RSD	
						ERR		ERR	<20% RSD	
						ERR		ERR	<20% RSD	
						ERR		ERR	<20% RSD	
						ERR		ERR	<20% RSD	
						ERR		ERR	<20% RSD	
						ERR		ERR	<20% RSD	
						ERR		ERR	<20% RSD	
						ERR		ERR	<20% RSD	
						ERR		ERR	<20% RSD	
						ERR		ERR	<20% RSD	
						ERR		ERR	<20% RSD	
						ERR		ERR	<20% RSD	
						ERR		ERR	<20% RSD	
						ERR		ERR	<20% RSD	
						ERR		ERR	<20% RSD	
						ERR		ERR	<20% RSD	

ATTACHMENT 8

Certification Statement (IDC)

Demonstration of Capability
Certification Statement

Page __of __

il, air, solid, biological tissue)
and Analyte, or Class of Analytes or Measured

etals by 6010, benzene by 8021, etc.)

that:

ove, using the cited test method(s), which is in use at
amples under the National Environmental Laboratory
et the Demonstration of Capability.

performed by the analyst(s) identified on this certification.

d(s) and the laboratory-specific SOPs are available for all

the demonstration capability are true, accurate, complete

copy of this certification form) necessary to reconstruct
ve been retained at the facility, and that the associated
nd available for review by authorized assessors.

	Signature	Date
	Signature	Date

SOUTHWEST LABORATORY OF OKLAHOMA, INC. *and*

AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.

Standard Operating Procedure

Laboratory Corrective Action

Document No.: SWL-GA-105

Rev. No. / Date: Rev. 4.0 — 09/10/03

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APPROVALS	DATES
Melissa Crowder Procedure Prepared By	9-12-03 Date
Robert W. Mathis Laboratory Manager or Director	9/12/03 Date
Chuck Bonner Laboratory Safety Officer	9/11/03 Date
Chuck Bonner Laboratory QA/QC Officer	9/11/03 Date

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

Document Status

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

[✓] Official Copy OC No.: 2611 Issued to: TT NUS-PA Date: 10/17/03 Initials: [illegible]

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LABORATORY CORRECTIVE ACTION

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1.0 PURPOSE AND APPLICATION

This document defines the Corrective Action Program employed by Southwest Laboratory of Oklahoma, Inc. It delineates Corrective Action (CA) procedures including when CA is needed, how to document CA, and who is responsible for each step of the process. In addition, CA is distinguished from Non-conformance (NC). Procedures invoked for non-conformances are also described.

1.1 Corrective Action

CA is the step-wise process by which the laboratory identifies deficiencies, defines cause, and corrects deficiencies. It is an ongoing process by which laboratory operations are improved. Any condition determined to adversely affect quality shall be subject to CA.

CA is required when a laboratory system is deemed out-of-control, thus affecting the usability or quality of that system. This includes analytical and non-analytical systems. Corrective action is also required when an uncontrolled deviation from standard operating procedure is made. CA may be initiated by any laboratory personnel but should be reviewed by immediate supervisor.

1.2 Non-conformances

Non-conformances are minor, non-trending deviations from standard procedure or QC acceptance criteria which may cause reduction of future quality if trends form.

Events requiring non-conformance reports (NCRs) generally involve re-preparation or re-extraction of a sample. A NCR is documentation of action taken when a system non-conformance is identified. A system non-conformance is not considered an out-of-control event and does not affect an entire system (usability or quality). Trends of non-conformance, however, do constitute out-of-control events.

2.0 RESPONSIBILITIES

2.1 Corrective Actions

Responsibility for each Series of the CA process is delineated in Figure 1. G-Series CAs are the responsibility of the Laboratory Director and/or Laboratory Manager. The O-, I-, and R-Series CAs are the responsibility of the Program Managers of their respective sections. Responsibility for subcategories of the O-, I-, and R-Series lies with the Section Supervisors. G-Series, as described above, encompasses many different sections. Supervisors/managers of these sections will be responsible for their respective CAs (e.g., Client complaint CAs will be implemented through the Project Officers). In all instances, final follow-up and approval is the responsibility of the QA Department Manager. Portions of the CA process may be delegated, however, ultimate responsibility does remain with the parties described above.

2.2 Non-Conformances

2.2.1 NCRs are generally initiated at the department level and are the responsibility of the appropriate Section Supervisor or Program Manager. Actions to be taken, such as re-preparation or re-extraction, are the responsibility of the Section Supervisor performing the re-preparation or re-extraction. Section Supervisors will pull closed files and search for trends on a quarterly basis.

2.2.2 It is the responsibility of the Analyst to report Non-Conformances to their Supervisor &/or Program Manager.

2.2.3 It is the responsibility of the Supervisor &/or Program Manager to alert the Project Officer of the Non-Conformance.

2.2.4 It is the responsibility of the Project Officer to Notify the client and document the notification either via e-mail message or a Laboratory/Client Communication Form. This documentation becomes part of the case file and is included in the final report.

3.0 EQUIPMENT

Not Applicable

4.0 PRECAUTIONS

Not Applicable

5.0 PROCEDURE

Identify the problem using resources derived for training and SOPs. If at the bench level, then notify Section Supervisor of possible out-of-control event. Initiator of action and Section Supervisor or appropriate Manager will use the following guidelines to distinguish between the need for the CA or NC process and determine the action to be taken.

5.1 Corrective Action

Out-of-control events (See definitions – Section 9.5) require CA. Out-of-control events may include but are not limited to:

- Statistical outliers (as defined in the Quality Assurance Manual);
- Failure to follow standard operating procedure;
- System failure;
- Atypical instrument failure.

NOTE: If an out-of-control event is revealed, all actions associated with the out-of-control event must be stopped until the root cause is determined and CA is taken and documented.

Structure of the CA process is divided into two major sections or series: G-Series; and O-, I-, and R-series. The G-series CAs are those CAs which universally affect the laboratory such as Safety, Document control, Waste management, Sample log-in, Administration, Client Services, Purchasing, and Computer Services. The O-, I-, and R-series are general

laboratory sections: Organic, Inorganic, and Radiation Sections, respectively. Subcategories may be found under the second level/series to further differentiate between individual departments for filing and follow-up purposes (e.g., O-GCMS for Mass Spectrometry Department).

If it is decided that CA is required, then:

- Obtain a copy of the Corrective Action Report (CAR) form [See Attachment 1—Corrective Action Report];
- Fill out the CAR beginning with out-of-control event description, date of occurrence, name of CA initiator, samples/systems affected, Series, and Department.

  Documentation of CA (See Figure 2 for general Flow Diagram of Documentation Process):

  - Describe events including root cause,
  - Identify affected samples for analytical events,
  - Describe resolution,
  - Describe return to analytical control (proof of effectiveness),
  - Describe follow-up to prevent recurrences, and
  - Describe responsibility assignments.
- Copy initiated CA to the QA Department's OPEN file. The original copy is kept at the Responsible Party's Level in the OPEN file.

5.1.1 Define cause, including root cause, of out-of-control event:

5.1.1.1 Defining cause may be as simple as describing the event (e.g., mis-dilution of a sample because of a misunderstanding of procedure). Root cause describes why the sample was mis-diluted (e.g., needs more thorough training to understand method requirements). Both cause and root cause must be detailed in the CAR in the appropriate section.

5.1.1.2 Root causes of out-of-control events may be separated into six general categories which include:

- Need for organization,
- Need for training,
- Need for discipline,
- Need for resources,
- Need for time, and
- Need for top management support.

5.1.1.3 The following documentation must be made on the CAR at this point:

- Detail the cause and root cause of the event on the CAR in the appropriately marked section.

5.1.2 Define the need for change in the system, if necessary, and specify the changes to be made:

5.1.2.1 After determination of cause and root cause, the Responsible Party must determine whether the event warrants change in a system, SOP, and/or QA manual. A change must be made if the system, SOP, etc., might perpetuate the out-of-control event.

5.1.2.2 All changes in procedure, accompanied by change in documentation such as SOPs, QA Manual, etc., must be approved by the QA Department Manager. The QA Department Manager is responsible for notifying other affected Sections of the change.

5.1.2.3 Documentation must be made on the CAR:

- Note on the CAR if changes must be made, the changes to be implemented, and the accompanying documentation changes that must be made;
- Signatures and dates documenting Responsible Party and QA Manager's approval must be made on the CAR at this time.

5.1.3 Plan of action with implementation schedule:

5.1.3.1 After changes to be made have been determined, a plan of action with schedule must be documented on the CAR.

5.1.3.2 Documentation must be made on the CAR:

- Step-by-step plan of action to implement the necessary change;
- Projected date of completion of the change.

5.1.4 Implementation:

5.1.4.1 Follow the plan of action, making every effort to meet the projected schedule of completion. Completion includes procedural change, documentation change, re-training of affected personnel, and client notification (if applicable).

5.1.4.2 Upon completion of the change:

- the Responsible Party will sign and date the appropriate section of the CAR.

5.1.5 Follow-up to ensure change is made and to assess effectiveness of change:

5.1.5.1 Follow-up by Responsible Party includes audits of: effectiveness of the procedural change, documentation of the procedural change, and documentation of appropriate re-training.

Documentation to be made on the CAR:

- Describe the follow-up procedure;
- Note the effectiveness of the change;
- Signature and date of the Responsible Party's follow-up completion.

Upon completion of follow-up:

- The Responsible Party will sign and date the appropriate section of the CAR;
- Original copy of the CAR will be transferred to the QA Department. Responsible Party will file a copy of the CAR in his/her CLOSED file.
- If follow-up reveals that the change was not effective, then the CA process must be repeated starting at section 8.1.2.

5.1.5.2 Follow-up by QA Department is the final step in the CA process. Follow-up includes audits in the following areas: effectiveness of the procedural change, documentation of the procedural change, and documentation of appropriate re-training.

5.1.5.3 Upon completion of QA follow-up:

- The QA Manager will sign and date the appropriate section of the CAR, documenting that follow-up was performed;
- Original copy of the CAR is transferred to the QA Department CLOSED file.
- If follow-up reveals that the change was not effective, then the CA process must be repeated by the Responsible Party starting at section 8.1.2.

5.1.6 Quarterly review of Closed CAs to search for trends:

5.1.6.1 Both the QA Department and individual Laboratory Sections must quarterly review all Closed CARs to search for trends.

5.1.6.2 Each CAR reviewed must be initialed and dated to document that the review was performed.

5.2 Non-conformance

Non-conformances require that a NCR be completed. Non-conforming events include minor, non-trending deviations from standard procedure or QC acceptance criteria which may cause reduction of future quality if trends form. Events requiring a NCR generally involve re-preparation or re-extraction of a sample. Incidents such as broken samples and/or reagents are reported to QA through the NCR process and tracked through this mechanism.

NCR Process:

- Identify and assess the problem event (out-of-control or non- conformance).
- Define cause of the non-conformance.
- Define the action to be taken.
- Quarterly review of NCRs to search for trends (discovery of trends will constitute the need for CA).

NCR documentation:

- Describe events,
- Identify affected samples for analytical events,
- Describe action to be taken,
- Describe responsibility assignments.

If a decision is made that a NC has occurred then:

5.2.1 Obtain a copy of the Non-conformance Report (NCR) form [See Attachment 2 – Non-conformance Report].

5.2.2 Fill out the NCR beginning with the department affected, Section Supervisor, the name of initiator, date of occurrence, description of the event, and the samples affected.

5.2.3 Define the cause of non-conformance:

5.2.3.1 Defining cause may be as simple as describing the event (e.g., erratic QC recovery caused by poor technique). Cause and action to be taken must be detailed in the NCR in the appropriate section. The "Cause" portion of the report will be signed by the Section Supervisor.

5.2.3.2 Copy the initiated NCR to the department performing the re-preparation or re-extraction. The original copy is kept by the initiator's Department.

5.2.3.3 If re-preparation or re-extraction is required, the NCR must be signed and dated upon completion by the Section Supervisor who is responsible for re-preparation/re-extraction.

5.2.4 Check for NC trends by periodic audit of files. If trends are noted, CA must be initiated. Section Supervisors or appropriate managers are responsible for NCR follow-up.

- Documentation to be made on the NCR:

Section Supervisor or appropriate manager will initial and date the NCRs, which were reviewed upon completion.

5.2.5 Non-Conformances will be forwarded to the Project Officer and client(s).

6.0 RECORDS

6.1 Corrective Action Report

6.2 Non-conformance Report

7.0 REFERENCES

7.1 Navy Installation Restoration Laboratory Quality Assurance Guide, February 1996.

7.2 Scope of Work for Analytical Sources, April 1996, Department of Energy. Page 50-51.

7.3 Alan J. Sayle, Management Audits, the assessment of quality management systems, 2nd Edition, ASQC Quality Press, Copyright 1988. Page 1-5.

8.0 DEFINITIONS

Corrective Action (CA) – the step-wise process by which laboratory deficiencies are identified, causes are defined, deficiencies corrected, and possibility or recurrence minimized and is an ongoing process by which laboratory operations are improved. Corrective action is required when a laboratory system is deemed out-of-control, thus affecting the usability or quality of that system. This includes analytical and non-analytical systems.

Corrective Action Report (CAR) – documentation of CA with emphasis on describing events, root causes, affected samples, resolutions including return to analytical control, and follow-up for recurrence prevention.

Closed File – a closed file is a file containing a CA that has been completed through final Follow-up by the QA Department.

Open File – the open file is a file containing a CA which is still in-process.

Out-of-control event – an event which is contrary to or fails to meet the objectives of the laboratory QA/QC plan, laboratory SOPs, or good laboratory practice thus affecting the usability or quality of a system. This includes analytical and non-analytical systems.

N/A – not applicable.

Non-conformance (NC) – non-conformances are minor, non-trending deviations from standard procedure or QC acceptance criteria which may cause reduction of future quality if trends form. Events requiring NC generally involve re-preparation or re-extraction of a sample.

Non-conformance Report (NCR) – documentation of action taken when a system non-conformance is identified. A system non-conformance is not considered an out-of-control event and does not affect an entire system (usability or quality). Trends of non-conformance, however, do constitute out-of-control events.

Responsible Party – the responsible party is the individual who is responsible for seeing the corrective action process through to closure. See Section 2.1.5 and Figure 1 for details.

9.0 ATTACHMENT

9.1 Figures (Immediately Following Text)

9.1.1 Corrective Action Report (CAR) Structure

9.1.2 Documentation Trail

9.2 Attachments (Immediately Following Text)

9.2.1 Attachment 1: Corrective Action Report

9.2.2 Attachment 2: Non-conformance Report

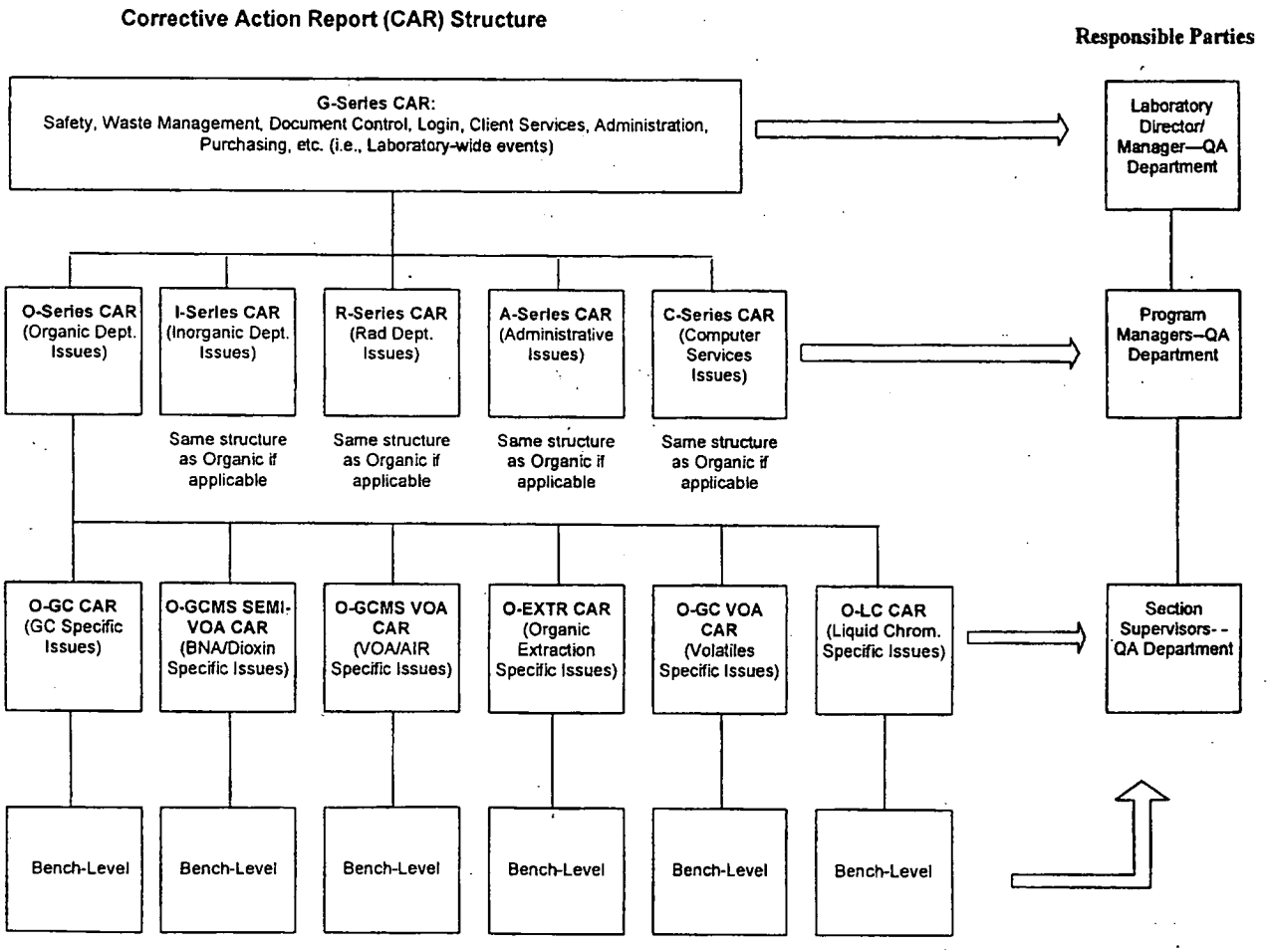

FIGURE 1: Corrective Action Report (CAR) Structure

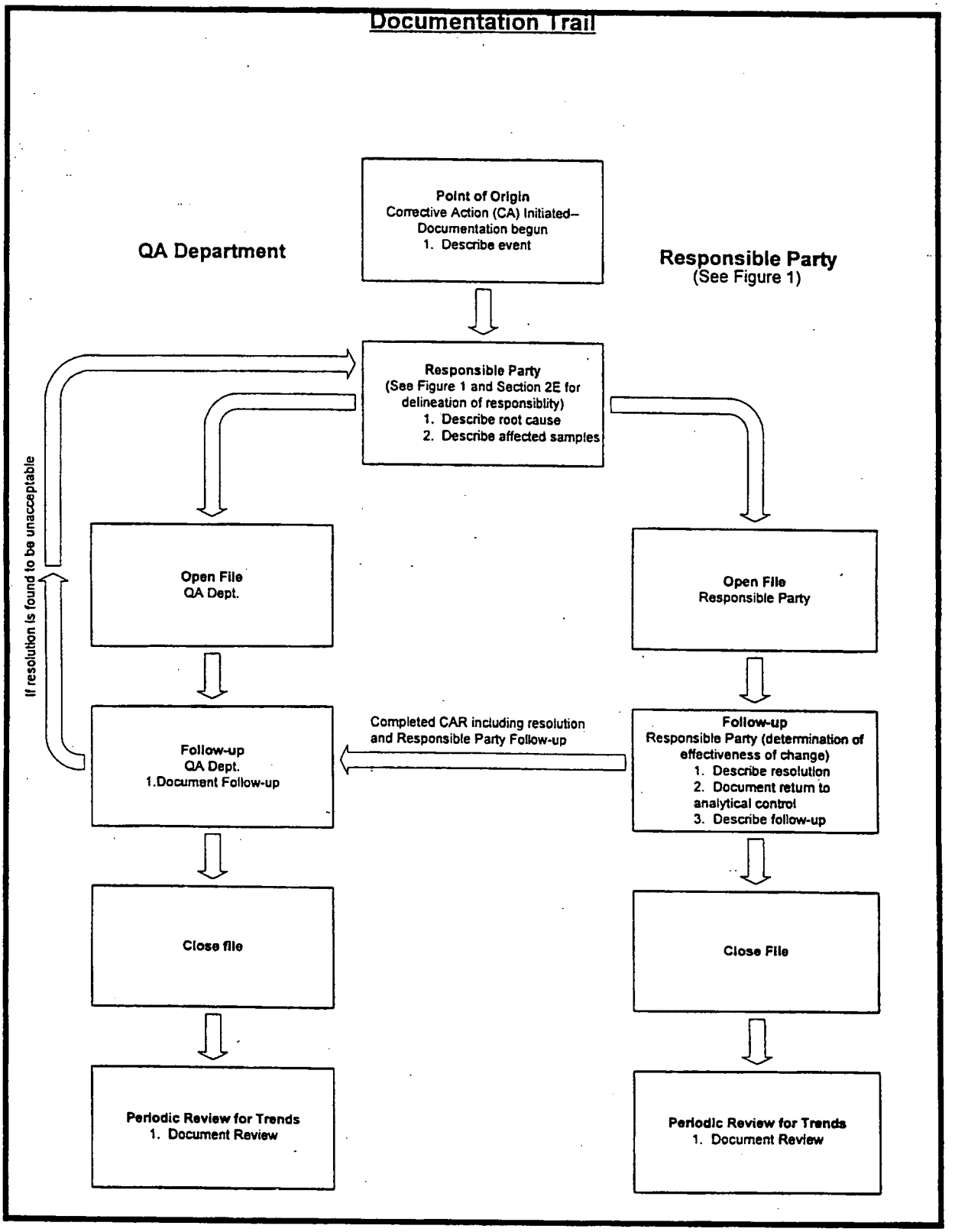

FIGURE 2: Documentation Trail

ATTACHMENT 1
Corrective Action Report

Corrective Action Report

SOP SWL-GA-105, REV. # 4.0, 09/10/03 FILE ID: GA105a1.doc

Series (Circle one): G, I, O, R **Department:** _____

Description of Out-of-Control Event

INITIATED BY: _____ DATE: _____
RESPONSIBLE PARTY: _____ DATE: _____
DATE OF OCCURRENCE: _____
DESCRIPTION OF EVENT:

SYSTEM AND/OR SAMPLES AFFECTED:

Cause/Root Cause

DESCRIPTION:

System Changes Required (Circle One): Yes / No

PROCEDURAL CHANGE DESCRIPTION:

DOCUMENTATION CHANGE DESCRIPTION:

SIGNATURES: Responsible Party: _____ Date: _____
QA Manager: _____ Date: _____

Corrective Action Report

SOP SWL-GA-105, REV. # 4.0, 09/10/03 FILE ID: GA105a1.doc

Continued from Page 1

Plan of Action

STEP-BY-STEP DESCRIPTION:

PROJECTED DATE OF COMPLETION: _____

Implementation

SIGNATURE: Responsible Party: _____ Date: _____

Departmental Follow Up

DESCRIPTION OF FOLLOW UP:

WAS ACTION EFFECTIVE?: YES / NO

CLIENT NOTIED? (IF APPLICABLE) YES / NO

SIGNATURE: Responsible Party: _____ Date: _____

QA Follow Up

DESCRIPTION OF FOLLOW UP:

WAS ACTION EFFECTIVE?: YES / NO

SIGNATURE: QA Manager: _____ Date: _____

ATTACHMENT 2
Non-Conformance Report

Non-Conformance Report

SOP SWL-GA-105, REV. # 4..0, 09/10/03 FILE ID: GA105a2.doc

Department: _____

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):

_____

_____

_____

Cause

DESCRIPTION:

_____

_____

_____

SIGNATURE: Section Supervisor: _____ Date: _____

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):

_____

_____

CLIENT NOTIED? (IF APPLICABLE) YES / NO

SIGNATURE: Section Supervisor: _____ Completion Date: _____

QA FOLLOW-UP

DESCRIPTION OF FOLLOW-UP:

_____

_____

_____

WAS ACTION EFFECTIVE?: YES / NO

SIGNATURE: QA Manager: _____ Date: _____

SOUTHWEST LABORATORY OF OKLAHOMA, INC. *and*
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TM

Standard Operating Procedure

Archival and Retrieval of Sample Documentation

Document No.: SWL-GA-106

Rev No. / Date: Rev. 3.0 — 05/28/02

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APPROVALS	DATES
Susan S. Rue Procedure Prepared By	May 28, 2002 Date
Robert W. Mathy Laboratory Manager or Director	5/30/2002 Date
Chuck Jones Laboratory Safety Officer	5/28/02 Date
Chuck Jones Laboratory QA/QC Officer	5/28/02 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.: 2611 Issued to: TT NUS-PA Date: 10/17/03 QA Initials: CAH

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ARCHIVAL AND RETRIEVAL OF SAMPLE DOCUMENTATION

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Attachment 1: Archival Inventory Record 8
Attachment 2: Archives Shelving – Signage 9

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/short term storage set forth in Southwest Laboratory of Oklahoma's (SWL's) "*Preparation, Review, Revision, and Control of Procedural Documents*," 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. 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 data. Accountability for this data, however, will remain with the Manager or Supervisor.

2.3 Each department's designated person shall record all filed data on the Archival Inventory Record form or similar form (see Attachment 1). The designee shall submit a copy of this record to the DCO (Document Control Officer) prior to transferring boxes of data to warehouse for long-term storage.

2.4 The DCO shall maintain single copies of the Archival Inventory Record mentioned in Section 2.3, in both hardcopy and database form.

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, SWL-GA-101 "*Preparation, Review, Revision, and Control of Procedural Documents*," and "*Archival and Retrieval of Sample Documentation*," SWL-GA-106.

2.6 It is the responsibility of the Marketing Department and the Project Officer (PO) to communicate special client needs regarding storage, archival, return, and disposal of data.

3.0 EQUIPMENT

Not applicable.

4.0 PRECAUTIONS

Not applicable.

5.0 PROCEDURES

5.1 General Data Storage Information

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

Laboratory data is generated, copied, and stored in boxes within each department. Once these boxes are full, data clerks log the contents of the boxes on an inventory sheet either exactly like or similar to Attachment 1, and then label the boxes (please see Section 5.1.1, or 5.1.2 for specific labeling requirements). The boxes are stored in each department storage area throughout the year and are moved to the hallway when more space is needed. They are kept in this location for six months to one year. On an annual basis, usually in the Fall or Spring the data is archived to the warehouse for long-term storage (five years for most clients; a shorter or longer period for clients who specify this at the on-set of the project). It is the data clerks responsibility to advise the Document Control Officer and team as to which boxes should be taken to the warehouse, and which should remain in the laboratory.

Prior to transferring boxes to the warehouse for archiving, an inventory sheet should be filled out for each box. The original sheet shall be used to make two additional copies: one sheet must be placed in data box (or in a transparent pouch attached to the front of the box); one sheet must remain with data clerks; and one sheet must be submitted to the Document Control Officer. In addition, each box must be properly labeled with the minimum amount of information as specified in Section 5.1.1, or 5.1.2.

Note: The Project Officer is responsible for advising the data clerks and the invoicing clerk and the Document Control Officer of any client requests to hold data onsite or in the warehouse for a shorter or longer period of time than stated herein. The PO should notify these clerks of any special storage requirements at the time this information is first received. The data clerks and invoicing clerk must then indicate these special instructions on the front of the box. It must be clearly indicated so that the DCO and team can easily determine when the box is to be destroyed. *Example*: If the most recent data in the box is dated 07/31/01, and the client requires that we hold data for 8 years rather than 5years, the clerks must write 07/31/09 on the box in the space marked "**Destroy Date**," or labeled clearly if no space for the "**Destroy Date**" already exists.

5.1.1 For CLP data packages, all departments shall follow the procedures described in the following paragraphs for departmental data storage.

NOTE: All Organic CLP type data packages not normally stored in each department must be routed and stored in the GC/MS Department.

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 or any other functional storage box.

[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 assigned to it by a data clerk or other designee. This number should be the next sequential number in the list of boxes already numbered and/or archived. The

number should have a prefix of letters identifying the department from which the data was generated (i.e. Org-147, indicates Organic Department, 147th box). Figure 1 is an example of proper labeling.

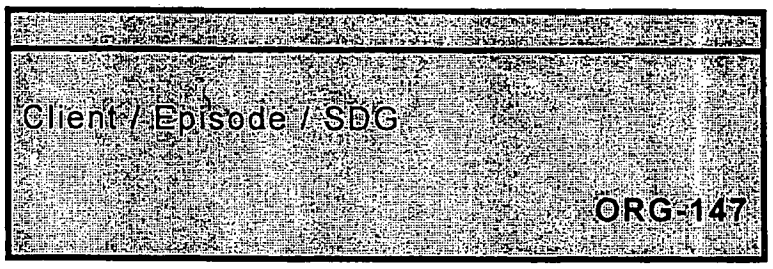

Figure 1: Laboratory Department Data Storage Box Identification

5.1.1.3 The Client Name, SDG #, and volume # (if any, i.e. Volume 1 of 3) of each must be labeled on the front of the box or written on a sheet of paper and placed in a plastic, transparent pouch attached to the front of the box. If the data does not belong to private clients, but are QA data, the box should be labeled accordingly (i.e. ICAL EPA, ICAL Private, etc.) Also, the analysis date (or report date) of the most recent data filed in the box must be labeled on the front cover. This date will determine when the boxes are to be destroyed. (Five years unless otherwise specified by client.) The "Destroy Date" must be indicated on the box and an inventory record (similar to or exactly Attachment 1, Archival Inventory Record) shall be completed and kept in a binder in the relevant department. The inventory record used, whether the version in Attachment 1 or one created by each department, must have the minimum information: Box ID, Archive or Inventory Date, Date of last entry, Department Name, Client Name, and SDG/Episodes.

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 section 5.2 for description of the offsite archival facility).

Note: Exceptions to this include any Arizona clients specifying an alternative onsite storage time of 2 years. See project specific SOPs for special requirements and details (SOP SWL-GP-203).

Prior to transferring boxes to the warehouse, each department designee shall confirm that any data taken out of the box is returned. Any 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 DCO who will keep all copies in a binder and in a database for each department. See Section 5.2 through 5.5 for the steps that should follow this section.

5.1.2 For non-CLP type reports (i.e.; standard tabular) all hardcopy data is routed to SWL's General Reporting Department. Once completed and prior to sending original report to clients, a copy is made for the laboratory and the copy is transferred to the Invoicing Department.

5.1.2.1 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. The transfer of data to the warehouse is done on an annual basis.

Note: Exceptions to this include any Arizona clients specifying an alternative onsite storage time of 2 years. See project specific SOPs for special requirements and details (SOP SWL-GP-203).

5.1.2.2 Boxes are identified as follows, for non-CLP reports (see Figure 2).

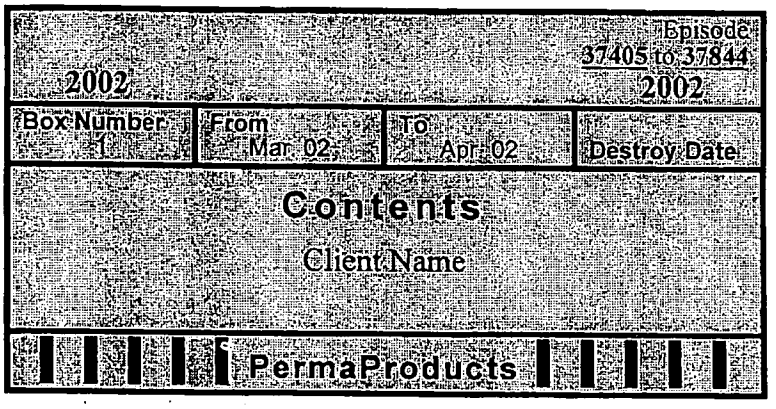

Figure 2: Reporting Dept. and Invoicing Storage

Episode = The range of SDG numbers from the smallest to the largest
2002 = The year(s) data begins and ends (which shall be labeled just above the "Box Number" and "Destroy Date"
1 = Box number for the Client for the whole year (i.e. 1 for first box, 2 for second, etc.)
Mar. 02 = Month and year taken from first episode/SDG in the box.
Apr. 02 = Month and year taken from last episode/SDG in the box.

NOTE: Invoicing Clerk will assign a "Destroy Date" of 5 years from date of most recent data in box, unless client specifications state otherwise.

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.)

5.1.2.3 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.

5.1.2.4 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 unless otherwise stated, at which time they will be transported to a predetermined paper recycling site.

5.1.2.5 *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 and Attachment 1 for example of form). Upon completion, two copies will be made: one copy is filed in the back of the box; one copy is submitted to the DCO; 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 1.5 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 Manager.

5.3 Archival of all Laboratory Logbooks

See SWL-GA-101, "*Preparation, Review, Revision, and Control of Documents*."

5.4 Final Disposal of Archived Data

Prior to transferring archive data from onsite storage to the warehouse, an assigned employee must first identify and remove all boxes in the warehouse that are 5 years old or more (unless otherwise noted). The Destroy Date should be clearly labeled on the front of the box. However, if it is not already labeled and if the data does not belong to a client with special archiving requirements, the 5-year rule applies. These boxes are to be taken by an established recycling company providing confidential services, to the predetermined recycling facility for disposal.

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. Special data storage or destruction will be noted on the front of the box by the data clerk or invoicing clerk (see section 5.1 for details).

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

5.5 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 DCO.

5.6 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 & 5.1.2.

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

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

The boxes will be separated by departments. For example, "OA_ _ _" boxes are "Air" data boxes and will be taken to the section of the warehouse where Air data are stored; "OD" are "Dioxin" data boxes and will be taken to the shelves containing Dioxin data, etc. Once grouped by department, the boxes will be put on the shelves in numerical order.

Inorganic Data	GC Data	Pesticide Data
↳ IN-1, IN-2	↳ OG-1, OG-2	↳ OP-1, OP-2

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

These boxes are grouped by year and then stacked in alphabetical order by client name.

i.e. 1995
↳ Misc. "A"
AATS

5.7 Data Retrieval

Any employee requesting data retrieval from the warehouse must submit a Retrieval of Data form (Attachment 2). The DCO then requests the warehouse keys from the Laboratory Manager.

5.7.1 The DCO must maintain the form and have the requester sign it again once all boxes, files, data, etc. are returned.

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 DCO will e-mail reminders to the requester on a weekly basis until all data is returned. All data must be returned to the DCO, who will re-archive them in the appropriate box/shelf in the warehouse.

5.7.2 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 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

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

8.0 DEFINITIONS

Not applicable.

9.0 ATTACHMENTS

Attachment 1: Archival Inventory Record

Attachment 2: Data Retrieval Form

ATTACHMENT 1
Archival Inventory Record

Department: _____

Box ID: _____

Box Year: _____

Program Mgr./Section Supervisor: _____

_____ *Original Inventory Performed by*

_____ *Original Inventory Date*

_____ *Inventory Verification Performed by*

_____ *Inventory Verification Date*

Client	SDG	Listing of Episode	Brief Description of Contents

ATTACHMENT 2
Data Retrieval Form

Data Retrieval Form, SWL-GA-106

GENERAL ADMINISTRATIVE

SOP SWL-GA-106, 05/28/02, 3.0 FILE ID: GA106a2.doc

Department: _____

Program Mgr./Section Supervisor: _____

_____ *Retrieval Requester*

_____ *Date*

_____ *DC Officer*

_____ *Approval Date*

_____ *Retrieval Requester return signature*

_____ *Return Date*

Year	Box ID	SDG	Listing of Episode	Brief Description of Contents Needed & Reason for Retrieval

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

_____

_____

_____

_____

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GA-108/-109/-110

The Standard Operating Procedures (SOPs) contained herein are adopted by Pace Analytical Services.

These SOPs will be followed until superceded by Pace Analytical Services SOPs.

Signature:	Signature:	Signature:
Chuck Hoover	Roger C. Splinter	Dave Neal
Quality Assurance Officer	Dir. of Quality, Safety, Training	General Manager
Pace Analytical Services, Inc.	Pace Analytical Services, Inc.	Pace Analytical Services, Inc.
Broken Arrow, Oklahoma	Minneaplolis, MN 55414	Lenexa, KS

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. 5.3 — 09/08/03

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APPROVALS	DATES
Melissa Gomez Procedure Prepared By	9-12-03 Date
Robert W Mathis Laboratory Manager or Director	9/12/03 Date
Chuck Toney Laboratory Safety Officer	9/9/03 Date
Chuck Toney Laboratory QA/QC Officer	9/9/03 Date

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

Document Status

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

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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

Not applicable

4.0 PRECAUTIONS

Not applicable

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 sample (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 department/areas audit, the personnel involved, 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 main 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(l) 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 des s 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 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 reported 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: CFH

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			X			X			X		
GA132	DATA REPORTING (NON-EPA)										X		
GA146	LIMS RESULTS ENTRY PROGRAM										X		
GA106	ARCHIVAL										X		
GA131	**PROJECT MANAGEMENT**			X									
GA-142	ANAYTICAL REQUEST FORMS			X									
GA-144	REVIEW OF PROPOSALS, BIDS, CONTR			X									
GA-145	HANDLING COMPLAINTS			X									
GA102	**INFO SYSTEMS**				X								
GA138	LIMS BACK-UP				X								
GA139	LIMS MAINTENANCE/MNGMNT				X								
GA104	**TRAINING**	X			X			X			X		
GA107	**PROCUREMENT**	X											
GA108	STD TRACEABILITY	X			X			X			X		
GA127	PREV. MAINTENANCE	X			X			X			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			X			X			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.				
GA100	THEMOMETER CALIB CHECK	X											
GA100	THEMOMETER ID CHECK				X			X			X		

SOUTHWEST LABORATORY OF OKLAHOMA, INC. *and*

AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.

TM

Standard Operating Procedure

Traceability of Standards

Document No.: SWL-GA-109

Rev No. / Date: Rev. 1.3 — 09/23/03

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APPROVALS	DATES
Melissa Chandler Procedure Prepared By	9-26-03 Date
Robert W. Harris Laboratory Manager or Director	9/26/03 Date
Chuck Hamer Laboratory Safety Officer	9/23/03 Date
Chuck Hamer Laboratory QA/QC Officer	9/23/03 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: _____

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TRACEABILITY OF STANDARDS

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1.0 PURPOSE AND APPLICATION

1.1 The purpose of this standard operating procedure is to provide guidelines for standard traceability throughout the laboratory. It will provide a basis for establishing a uniform system for standard traceability, inventory, and preparation.

1.2 Standard Operating Procedures (SOPs) and Log Forms will be prepared by each individual laboratory section following this guideline to maintain a consistent procedure for the handling of all standards received within the lab.

1.3 Vendors of supplied standards will be selected with care, utilizing reputation and past supply history in the decision to order their standards.

2.0 RESPONSIBILITIES

2.1 The Program Managers/Department Supervisors or their designee are responsible for the preparation of each SOP and Log Forms required within their section.

2.2 The laboratory staff within each section will be responsible for the tasks included in each SOP which include, but are not limited to documentation of standard inventory, verification and/or assignment of standard expiration dates, filing of standard certificates of analyses, and standard preparation.

3.0 EQUIPMENT

3.1 Standard Preparation and Storage: Required syringes, pipettes, balances, glassware, and refrigeration as referenced in each laboratory section's SOP.

3.2 Certificates of Analyses, Data Packets, and/or Material Safety Data Sheets (MSDS): File Cabinets and Notebook Binders

4.0 **PRECAUTIONS.** All standards, reagents, and solvents are treated as hazardous and exposure to them is minimized. Any spills that may occur are cleaned up immediately.

5.0 PROCEDURE

5.1 Reference materials, whenever possible, should be traceable to NIST or EPA standard reference material.

5.2 All standards used for analysis within each section of the laboratory must be logged into an inventory log upon receipt. At a minimum, this log should include the standard's identification, vendor, lot number, receipt date, expiration date, purity, and verification of certificate of analysis or data packet receipt. If a vendor does not provide an expiration date for a standard, an expiration date will be assigned at the time of receipt. The criteria employed for expiration date assignment will be determined within each individual laboratory section.

5.3 Standards must be stored and maintained according to information provided by the commercial vendors.

5.4 Standard certificates must be maintained on file within each laboratory section for easy retrieval and review.

5.5 All standards prepared for analysis within each section of the laboratory must be recorded into a standards preparation log or form. At a minimum, when applicable this log should include the standard's identification, vendor, lot number, expiration date, concentration level, solvent name, solvent lot number, preparation date, preparer's initials, and laboratory identification number.

5.6 Upon opening, all standards will be labeled with the date opened.

5.7 All standards will be clearly labeled. At a minimum, each label should include the standard name, laboratory identification number, date prepared, preparer's initials, expiration date, and solvent used for preparation.

5.8 The quality of prepared standards will be verified by each laboratory section in one of two ways: 1) The standard will be analyzed as a check standard prior to use, or 2) The standard will be compared on-line to previous standard performance (i.e.: absorption, area, response factor). Each section's SOP will dictate the procedure performed and determine acceptance criteria.

5.9 Expired standards are tagged and segregated from those that are current.

6.0 RECORDS

6.1 Inventory Log or Form--Maintained upon the receipt of all standards. (See Attachment 1)

6.2 Standard Preparation Log or Form--Maintained at the time of each individual standard preparation. (See Attachment 2)

6.3 Standard Files--Maintained to include the current Certificates of Analyses, Data Packets, and Material Safety Data Sheets (MSDS)

7.0 REFERENCES

7.1 *Laboratory Safety Plan*, SWL-GA-111.

7.2 *Hazardous Waste Management Plan*, SWL-GA-114.

8.0 ATTACHMENTS

8.1 Attachment 1: "GC Volatile Standards Inventory Log"

8.2 Attachment 2: "GC Volatiles Standard Log"

9.0 DEFINITIONS

9.1 Terminology

Standard Reference Material:..... NIST or EPA standards in which reference material (standards) are traceable to.

Reference Material:..... Standards traceable to NIST or EPA standards.

Neat Standard:..... A pure analyte obtained from a vendor.

Commercially Prepared Standard/Solution: A standard or solution prepared by a commercial vendor, normally at a relatively high concentration.

Stock Standard:..... A standard solution prepared at a concentration by dilution of a neat or commercially prepared standard. This standard is always diluted before use.

Intermediate Standard:..... A standard solution prepared at a concentration by dilution of a stock standard. This standard may or may not be the working standard.

Working Standard:..... A standard at the daily calibration concentration.

Standard Operating Procedure (SOP):..... Written narratives that describe step-by-step instructions and procedures specific to the laboratory.

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.

Inventory Log or Form:..... Formal record maintained of all standards received from commercial vendors within each laboratory section.

Standards Preparation Log or Form:..... Formal record maintained of standards prepared within each laboratory section.

Certificates of Analyses/Data Packets: ..... Certification for commercially prepared standards provided by the vendors.

Laboratory Identification Number: ..... Unique number assigned to each prepared standard.

9.2 Acronyms

EPA: ..... United States Environmental Protection Agency

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

ATTACHMENT 1
GC Volatile Standards Inventory Log

Typist: LLR

GC VOLATILE STANDARDS INVENTORY LOG

GC VOLATILES DEPARTMENT

Ref: SWL-OG-113 Rev. 4.0
Book ID: OG-113-INV-1

Page _____ of Book __1____

	Standard Name/Vendor	Purity/ Conc.	Lot #	Quantity	Date Received	Expiration Date	Cert. of Analysis (Y/N)	Data Packet (Y/N)	Init.
1									
2									
3									
4									
5									
6									
7									
8									
9									
10									
11									
12									
13									

SOUTHWEST LABORATORY OF OKLAHOMA, INC./AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.
10926 E. 55TH PL., TULSA, OKLAHOMA OFFICE (918)664-0387 FAX (918)663-7841
F:\COMMON\ORGANICS\GCINV.DOC

Verified: _____

ATTACHMENT 2
GC Volatiles Standard Log

TM

GC·VOLATILES STANDARD LOG

GC VOLATILES DEPT

Ref: SWL-OG-113 Rev. 1.0
Book ID # OG-113-STD-2

Solution No.:_____ Page _____ of Book __2_____

Solution Expiration Date:_____ Solution Name:_____

Dilution Solvent:_____ Date:_____ Analyst_____

VENDOR/STANDARD	EXPIRATION DATE	LOT #	BEGIN CONC. (units)	ALIQUOT (uL)	TOTAL VOL. (units)	FINAL CONC. (units)

SOUTHWEST LABORATORY OF OKLAHOMA, INC
1700 W. ALBANY, BROKEN ARROW, OKLAHOMA OFFICE (918)251-2858 FAX (918)251-2599
F:\COMMON\ORGANICS\VOAGCSTD.DOC

Verified_____

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. 4.1 — 10/18/02

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APPROVALS	DATES
Procedure Prepared By	Date 11/04/02
Laboratory Manager or Director	Date 11/4/02
Laboratory Safety Officer	Date 10/18/02
Laboratory QA/QC Officer	Date 10/18/02

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

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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 (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 Following organic sample preparation, samples extracts are transferred to the analytical instrument analysis laboratories. Extract transfers are documented on their perspective Extract Receipt Log (see Attachment 10).

5.4.2.9 Samples that are depleted or broken during the analytical process are recorded on the depleted bottle log (Attachment 8) and the Internal COC Form. Analyst are to notify (verbally and/or via email messages) the QA Officer and Project Officer alerting them of the breakages. 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 Plan*").

Drum numbers listed on the disposal forms are recorded on the manifest upon transportation to the waste incinerating company.

5.4.2.10 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.

5.4.2.11 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 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

SOUTHWEST LABORATORY OF OKLAHOMA, INC.
1700 West Albany • Broken Arrow, Oklahoma 74012-1421
Office: (918) 251-2858 • Fax (918) 251-2599

TM

SAMPLING FIRM	CLIENT CONTACT	PHONE NUMBER

SWLAB PROPOSAL NUMBER	PROJECT NAME

SAMPLER: *(Signature)*

CLIENT ID/LOCATION	DATE	TIME	COMP.	GRAB	MATRIX	NUMBER OF CONTAINERS	ANALYTICAL TESTS REQUESTED								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)*

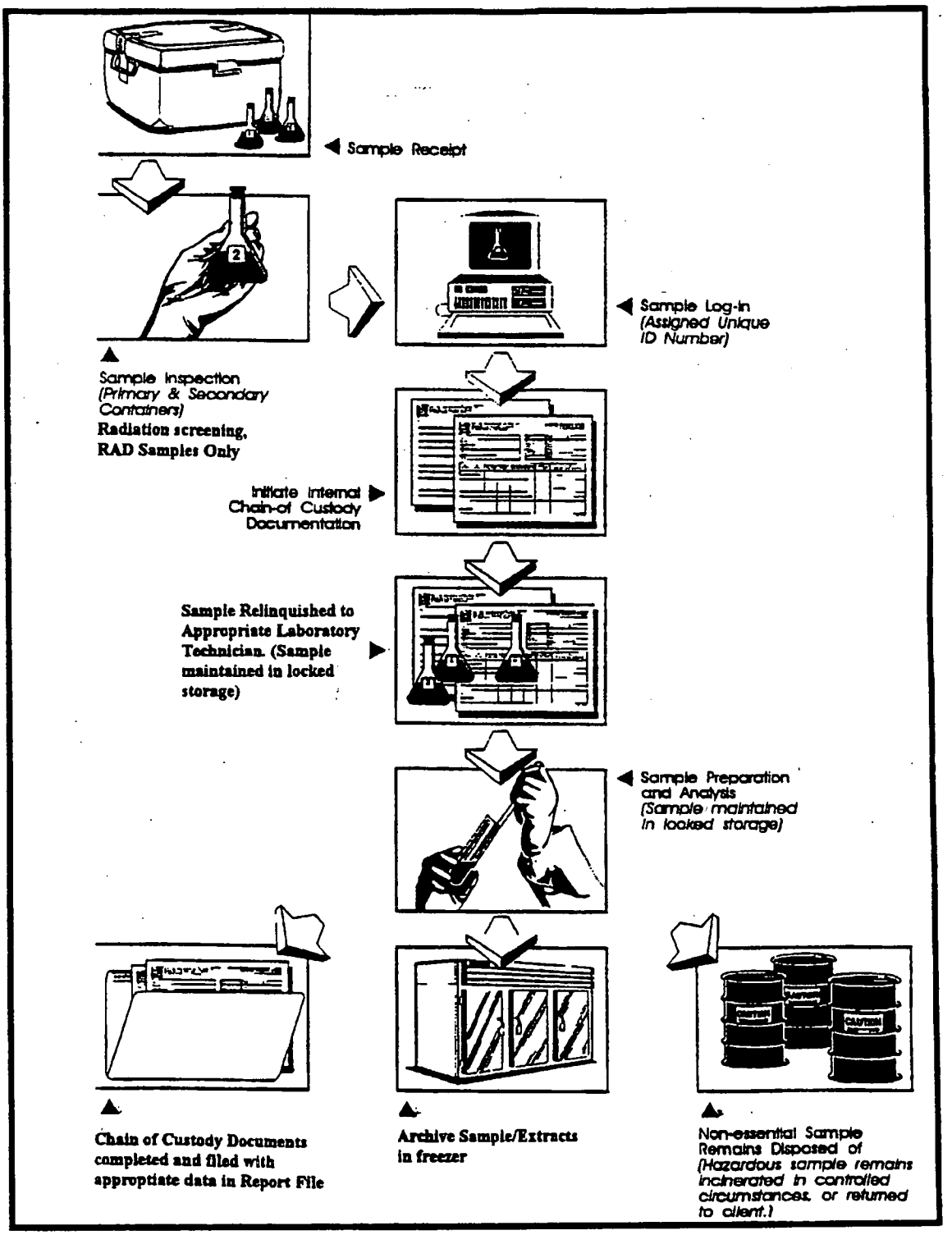
RELINQUISHED BY: *(Signature)*	DATE	TIME	RECEIVED BY: *(Signature)*

RELINQUISHED BY: *(Signature)*	DATE	TIME	RECEIVED FOR LABORATORY BY: *(Signature)*

REMARKS:

[SWL-GA-1104.0-09/13/01]

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	(Circle the Sample Numbers Checked out) #	#	#	#	#	#	#	Date/Time Out	Analyst	Date/Time In
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	8/24/00 0840	SR	8/24/00 1140
		8	9	10	11	12					
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	8/24 1400	SB	9/9 1450
		8	9	10	11	12	13				
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	AJC	8/28 500
		9	10	11	12						
MT620	C	1	2	3	4	6	7	8	8/28 9:00	SE	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]

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

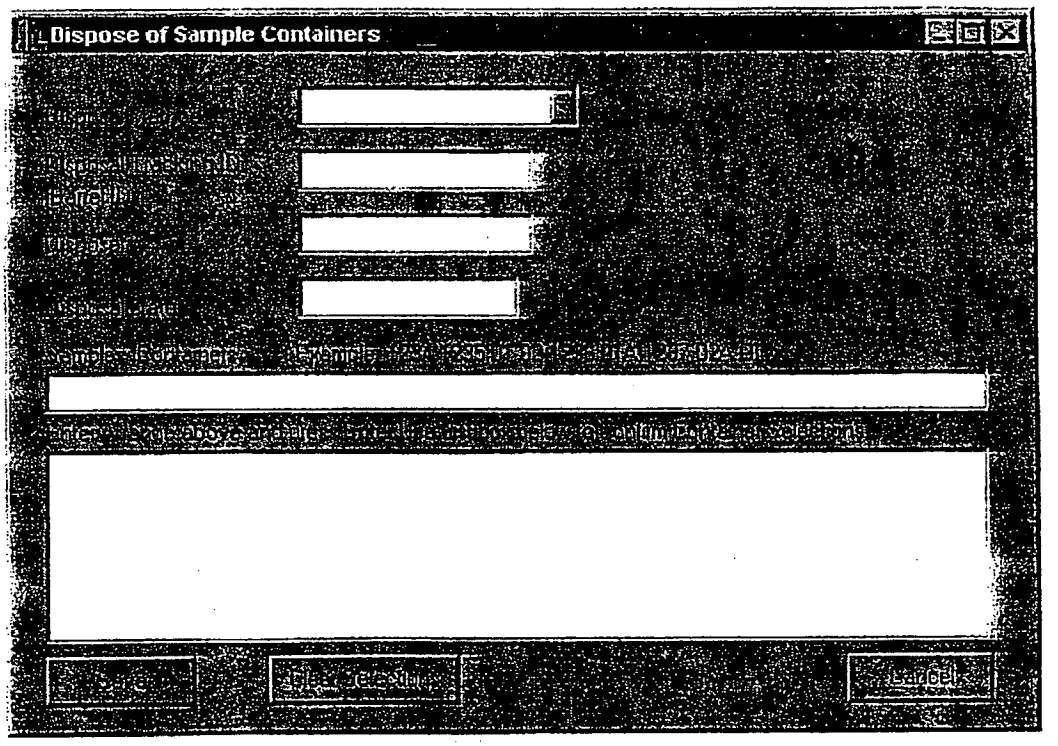
GENERAL ADMINISTRATIVE

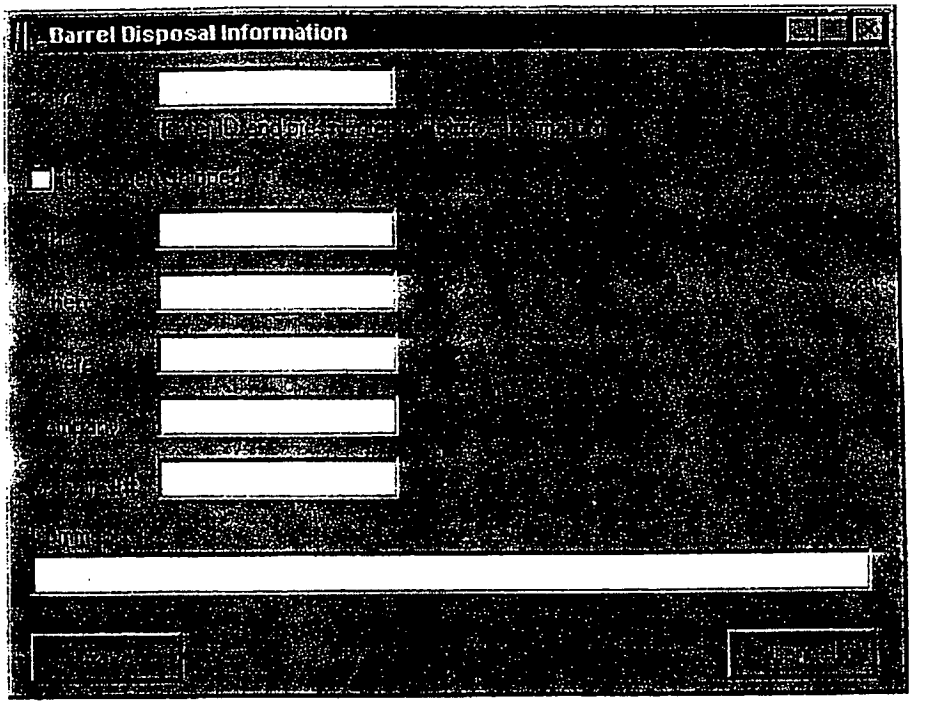
SOP SWL-GA-110, REV. # 3.2 FILE ID: GA110A8.doc Book # GA-110-PTCHBOTL

DATE	SAMPLE	REAGENT	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





ATTACHMENT 10
Extract Receipt Logs

Dioxin Extract Receipt Log, Ref: SWL-GA-110

Date	Time	Episode Number(S)	Client	Number of Samples	Extraction Chemist Initials	GC/MS Sample Custodian Initials	Archival Date

Pace Analytical®
www.pacelabs.com

Pace Analytical Services, Inc
1700 West Albany
Broken Arrow, OK 74012
Phone: 918.251.2858
Fax: 918.251.2599

GA-112 / -113

The Standard Operating Procedures (SOPs) contained herein are adopted by Pace Analytical Services.

These SOPs will be followed until superceded by Pace Analytical Services SOPs.

Signature:	Signature:	Signature:
Chuck Hoover	Roger C. Splinter	Dave Neal
Quality Assurance Officer	Dir. of Quality, Safety, Training	General Manager
Pace Analytical Services, Inc.	Pace Analytical Services, Inc.	Pace Analytical Services, Inc.
Broken Arrow, Oklahoma	Minneaplolis, MN 55414	Lenexa, KS

SOUTHWEST LABORATORY OF OKLAHOMA, INC. *and*

AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.

TM

Standard Operating Procedure

Control Charts

Document No.: SWL-GA-112
Rev No. / Date: Rev. 5.0 — 09/10/03

OFFICIAL COPY RED STAMP INDICATES ORIGINAL

APPROVALS	DATES
Melissa Ochoa Procedure Prepared By	9-12-03 Date
Robert W. Martin Laboratory Manager or Director	9/12/03 Date
Chuck Homer Laboratory Safety Officer	9/11/03 Date
Chuck Homer Laboratory QA/QC Officer	9/11/03 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.: 2651 Issued to: TT NUS - PA Date: 10/17/03 QA Initials: SAG

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CONTROL CHARTS

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1.0 PURPOSE AND APPLICATION

1.1 The performance of a measurement system can be demonstrated by the measurement of homogeneous and stable control samples in a planned repetitive process. The data generated is plotted in control charts to indicate whether the system is in control. It warns the laboratory of possible deviation from 95% confidence level by identifying systematic errors, drifts, or other types of problems.

1.2 There are two types of control charts used to monitor the laboratory data:

1.2.1 Laboratory Control Standard (LCS) Concentration vs. Date Analyzed (see Att. 1).

1.2.2 Percent Matrix Spike Recovery vs. Date Analyzed (see Att. 2).

1.3 The control chart mechanism is how SWLO develops internal standards.

1.4 For RAD Charts — In order to ensure that an instrument responds in the same manner subsequent to its calibration, an instrument response check is used in conjunction with an instrument control chart, known as an instrument tolerance chart. The response of an instrument control check source is directly linked to the calibration of the instrument. Therefore, if an instrument response check source becomes damaged or changes its characteristics, a new instrument control chart can only be established through the use of a new source, after verification that the instrument calibration has not changed. Otherwise, the instrument shall be recalibrated and a new instrument control chart established. Instrument response check sources should provide adequate counting statistics (less than a 1% Poisson statistic uncertainty) over the time period for which the source is to be counted. However, the source cannot be so radioactive as to cause pulse pileups, dead time that is significantly different from that to be expected from routine samples, or gain shift in the case of pulse height analyzer (PHA) systems. Any source to be used as a check source shall be either sealed or encapsulated to prevent loss of the source and contamination of the counting system which includes electroplated disks. The check source-to-detector geometry has to be known and held constant. The geometry is extremely important because small changes can have large effects that overshadow normal statistical counting variations.

2.0 RESPONSIBILITIES

2.1 It is the responsibility of each Department Head/Supervisor to generate the Control Charts for the Laboratory Control Standards (LCS/LCSD) and for the Matrix Spike Recovery (MS/MSD).

2.2 Review of the charts is also the responsibility of the Department Head/Supervisor.

2.3 Overseeing the Control Chart generation, investigation and corrective actions is the responsibility of the Quality Assurance Officer.

3.0 EQUIPMENT

3.1 Personal Computer, Gateway 2000, 486/33 processor or equivalent.

3.2 LIMS Server

- Borland® Quattro®Pro spreadsheet software Ver. 5.0, 1993 or Ver. 6.0, 1996.

4.0 PRECAUTIONS

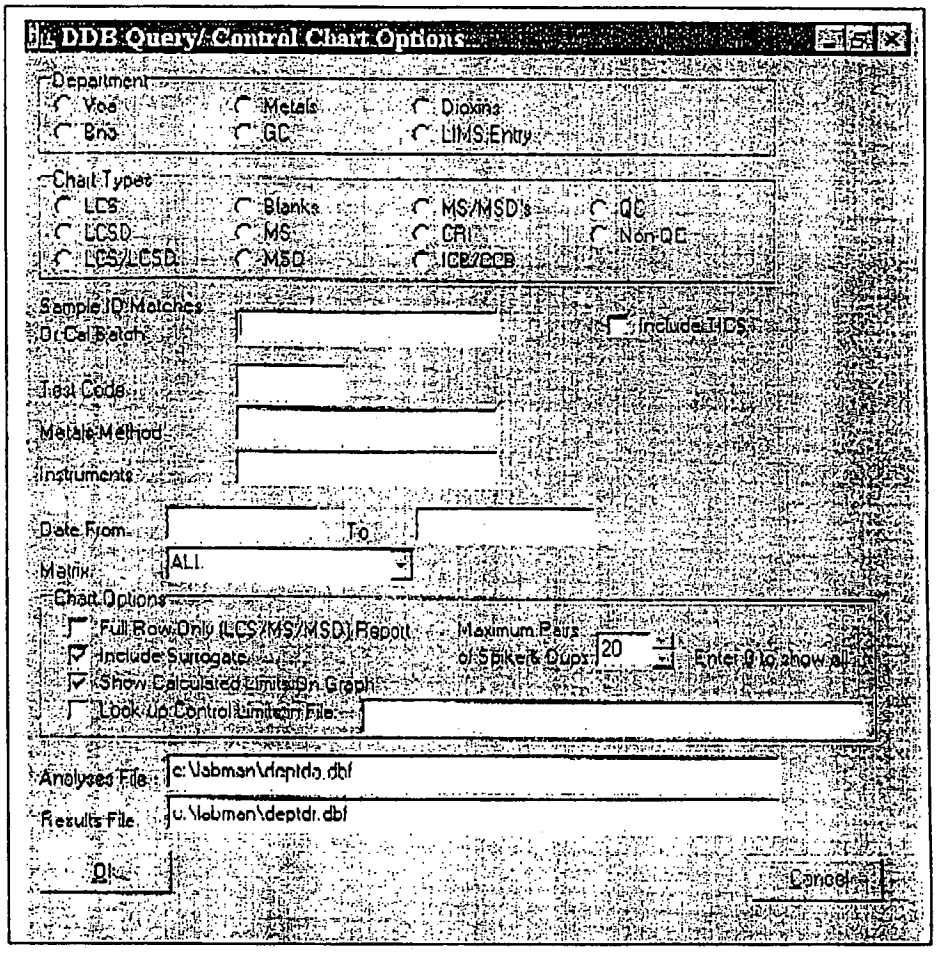
Not Applicable.

5.0 PROCEDURE

5.1 Data for control charts must be retrieved from the individual department databases by use of the "Shortcut to qcchart.exe" program.

Steps to generate a control chart are as follows:

5.1.1 DDB Query from the Control Chart pull-down menu — Direct/New Query.



5.1.1.1 Select the appropriate department database/chart type/test code/date range/matrix, etc.

5.1.1.2 The following fields are used as filters when requesting specific test and methods. They may be left blank when requesting all data.

Test code: VOA Lab MS300, MS313, MS399 etc.

BNA Lab: MS500, MS513, MS599 etc.

ICP Metals: Metals method must be entered

Metals Method: This applies only to ICP metals and sample types (LCS, MS, et. al.). SW846T is the only methodology option.

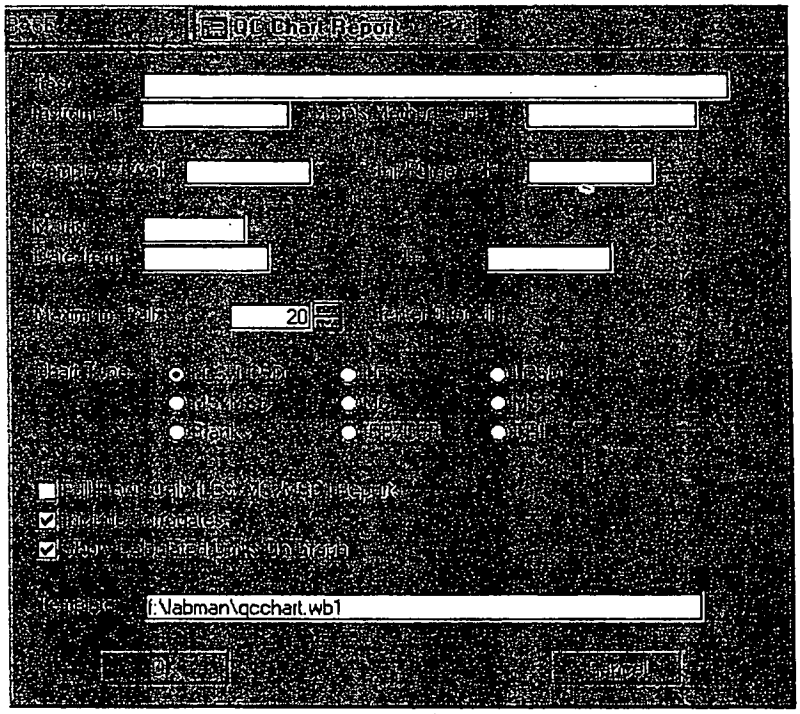
Date From/To: For BNA and ICP metals the prep. date is used. For VOA data enter the date analyzed.

5.1.2 Following the query, data is retrieved and presented in a spreadsheet.

To print the spreadsheet, right click in one of the upper cells without data. The menu allows the "print spreadsheet" option.

5.1.3 Viewing control charts.

To view the generated charts, click on one of the listed parameters. The associated chart is displayed with an option to print that particular chart or all charts associated with the spreadsheet.



5.1.4 When the data has been downloaded to the Quattro®Pro spreadsheet. The mean recoveries are calculated using the following:

$$X = \frac{\sum_{i=1}^{n} X_i}{n}$$

Where:

X_i, $i = 1$ to n are the percent recoveries from the n spike (LCS or MS) results and...

$\sum_{i=1}^{n}$ refers to the sum of the X values from $i = 1$ to n.

The standard deviation (S) of the data set is also calculated from the variance (S^2) using the following:

$$S^2 = \frac{1}{n-1}\left[\sum_{i=1}^{n} X_i^2 - \left(\sum_{i=1}^{n} X_i\right)^2 \Big/ n\right]$$

$$S = (S^2)^{1/2}$$

Where:

X_i, $i = 1$ to n, are the analytical results in the final method reporting units obtained from the n sample aliquots and Σ refers to the sum of the X values from $i = 1$ to n.

5.2 Control Chart Interpretation

5.2.1 For each parameter and method, a database of percent recovery (%R) for QC reference samples or spiked samples is collected. The arithmetic mean(x) and standard deviation(s) of this set is calculated. From this information, warning and control limits of a run are determined. They are defined as follows:

Warning limits: Defined as x ± 2s, where 's' is the Standard Deviation and 'x' is the Arithmetic Mean.

Control limits: Defined as x ± 3s, where 's' is the Standard Deviation. The percent recovery of each QC sample or spiked sample is plotted on a control chart and compared with the statistically based control limits.

Data Precision: Evaluated based on results of the samples analyzed in duplicate. The range (the difference between the spike and spike duplicate) is calculated and then divided by the average

of the two analyses, then multiplied by 100. This value equals the percent difference (%D). The mean percent difference of each data set is compared with the values previously found in the laboratory or in the method. The calculation for the control limit is +3s from the mean.

Precision charts are not generated at this time; However, the data precision calculations are part of the control chart spreadsheet.

Interpretation of control charts for data out of control:

- One or more points outside of the control limit (3s).
- A run of three consecutive points outside of the warning limits (2s).
- A run of eight points above or below 'x', indicating trends or shifts.
- Cycles or non-random patterns in the data chart (six consecutive points, increasing or decreasing).
- See Attachment 5 for examples of some of these outliers/trends.

Control charts are generated in the various departments where trends are noted and actions are taken to alleviate these patterns. Supervisors / Program Managers are to document their reviews on the charts, note any outliers / trends and initiate Corrective Actions (if needed).

5.2.2 Radiochemical Tolerance charts shall be interpreted objectively (see Attachments 6-8.) For a control chart, if a QC measurement occurs outside the expected three standard deviations statistical range, instrument service may or may not be needed. If the response of the instrument is not outside the established tolerance limits, the instrument may still be used. Depending upon the magnitude of the instrument response change, the operator may want to initiate an investigation into the root cause of the response change. To determine whether the outlier measurement was statistically significant, the analyst should run a series of repeated measurements and apply another statistical test, such as a Chi-square test, to determine whether the variation was excessive. Trends from control charts may show other information. For example, if regular measurements of the check source are moving in one direction, one can infer that some system variable is changing. This variation may not always require instrument service; instead, re-evaluation of the values of the standard deviation and other related limits of the control chart may be necessary. If the control chart has to be re-established, the calibration of the instrument or system has to be re-established or re-verified prior to the construction of a new control chart.

5.3 Policy on laboratory generated control limits.

5.3.1 Control limits will be used as follows:

5.3.1.1 Use of client specified control limits are outlined in the individual client's QAPP.

5.3.1.2 In the absence of client specified control limits, the laboratory will use in-house control limits.

5.3.2 LCS/LCSD — Data is downloaded from the LIMS system into a Quattro®Pro spreadsheet. Control charts are generated monthly. Statistical limits are derived from the previous year's data.

5.3.3 MS/MSD — Data is downloaded from the LIMS system into a Quattro®Pro spreadsheet. Control charts are generated monthly or quarterly (depending on the number of data points). Statistical limits are derived from the previous year's data.

6.0 RECORDS

6.1 Copies of Control Charts are maintained in each Section as well as in the QA Dept.

6.2 File listings and updated control limits are maintained in the Blue Book.

7.0 REFERENCES

American National Standard Measurement and Associated Instrumentation Quality Assurance, ANSI N42.23-1996.

8.0 DEFINITIONS

DDB:..... The laboratory's department specific databases. These databases contain results for the specific departments. The data requested is retrieved from these databases through networks.

Filter:..... Fields used to limit data on a report or query to specific values. When these fields are left blank, any value is acceptable.

Template: ..... A blank spreadsheet file used when Quattro ®Pro is initiated by the control chart's program.

LCS:..... Laboratory Control Sample. A water or soil blank sample (sand or glass beads) that is spiked with analytes of interest. The results for these analytes are carried through the analytical process to determine the efficiency of the analytical process.

MS:..... Matrix Spike. A sample spiked with analytes of interest to determine whether the matrix (water, soil, etc.) has an effect on the recovery of specified analytes when the sample is analyzed by a particular method.

Blue Book: .... A compilation of in-house generated Control Limits.

9.0 ATTACHMENTS

Attachment 1: Phenol LCS Recoveries

Attachment 2: Fluoranthene Matrix Spike

Attachment 3: ICP Metals Recoveries

Attachment 4: Copper Recoveries

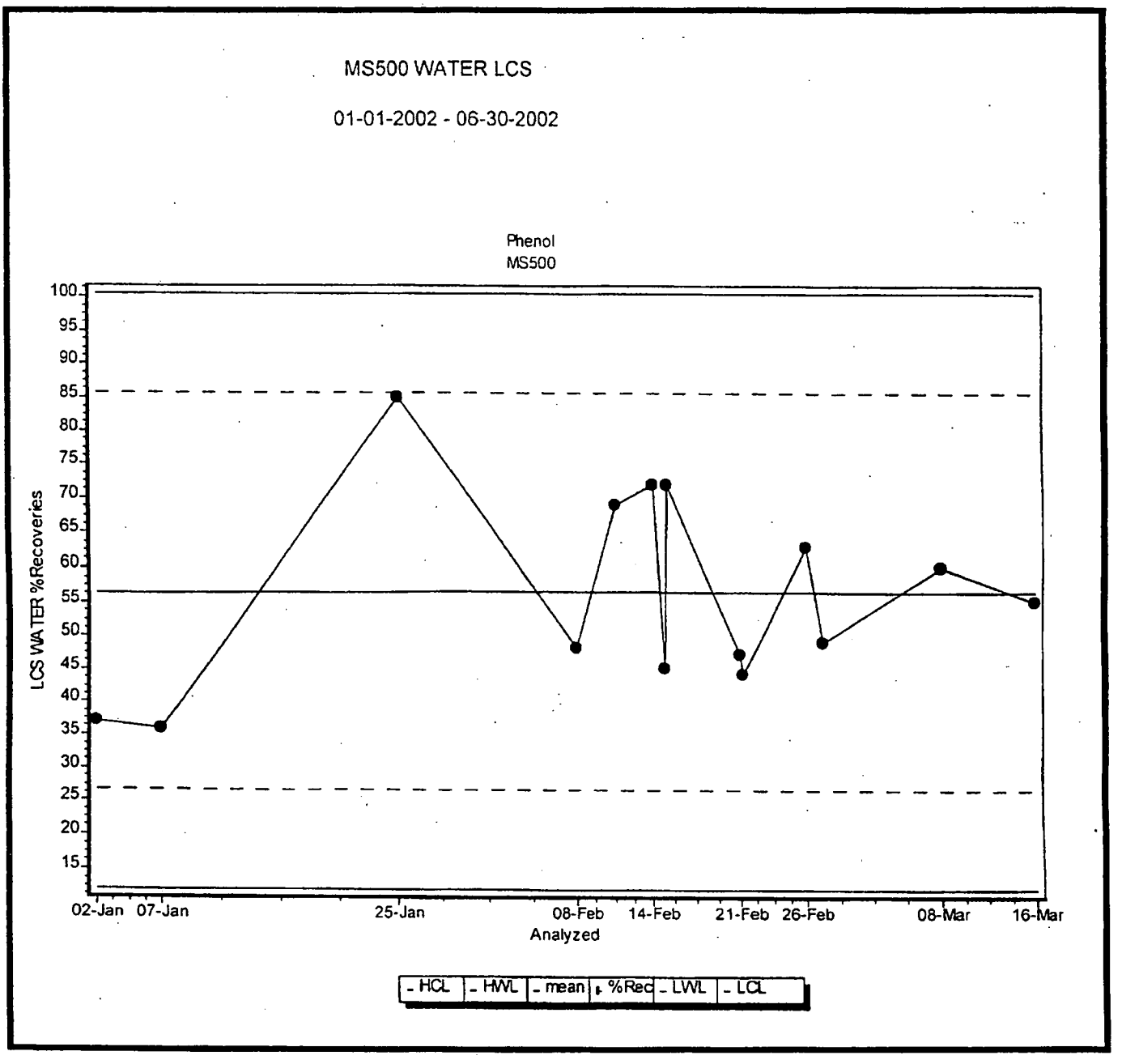
Attachment 5: 2,4-Dinitrotoluene Recoveries

Attachment 6: Radiochemical Tolerance Charts: LB-4000 A/B Source Check

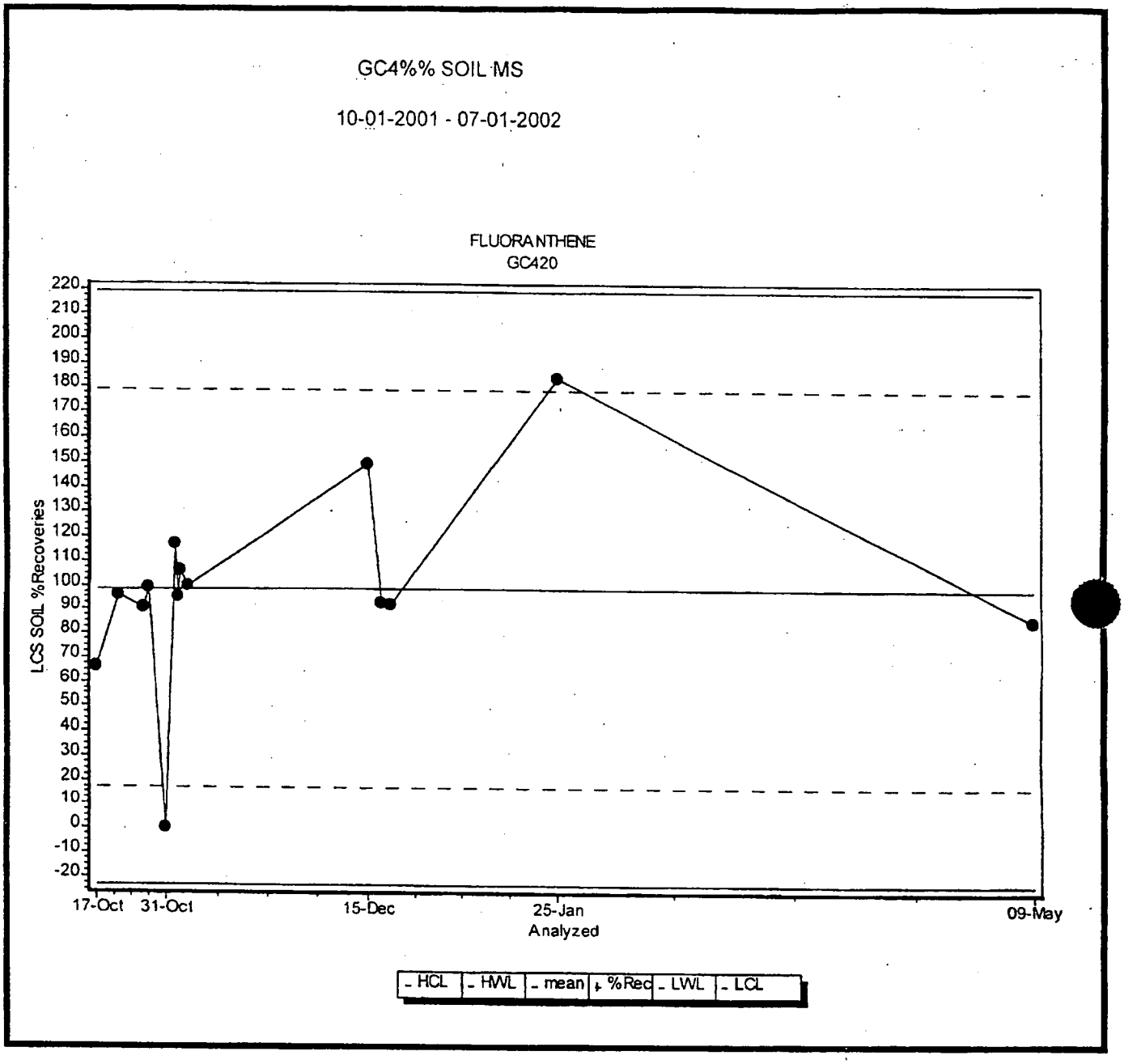
Attachment 7: Radiochemical Tolerance Charts: LB4100 A1-Alpha

Attachment 8: Radiochemical Tolerance (IPA) Charts

ATTACHMENT 1
Acenaphthene Recoveries



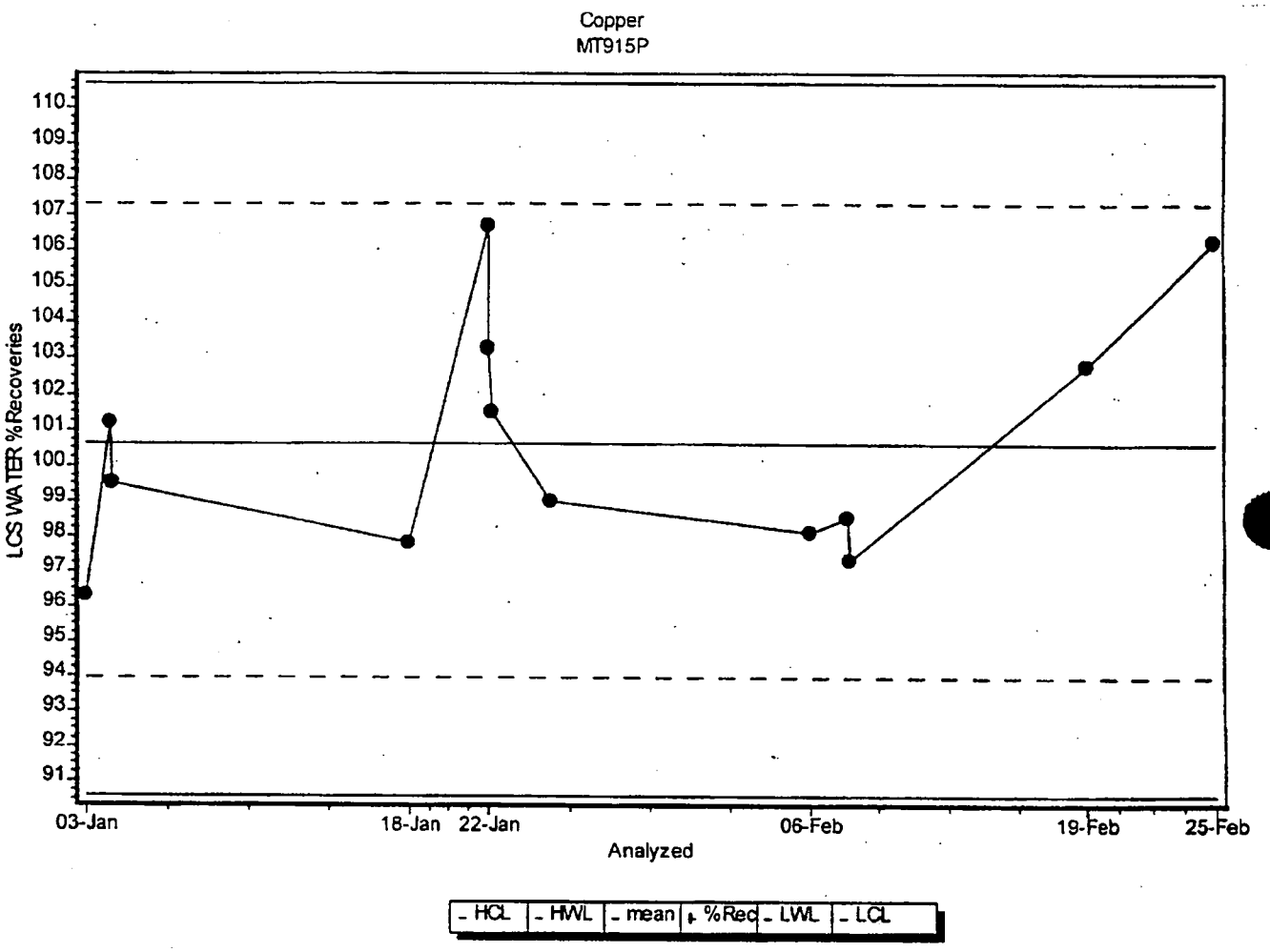
ATTACHMENT 2
Toluene Recoveries



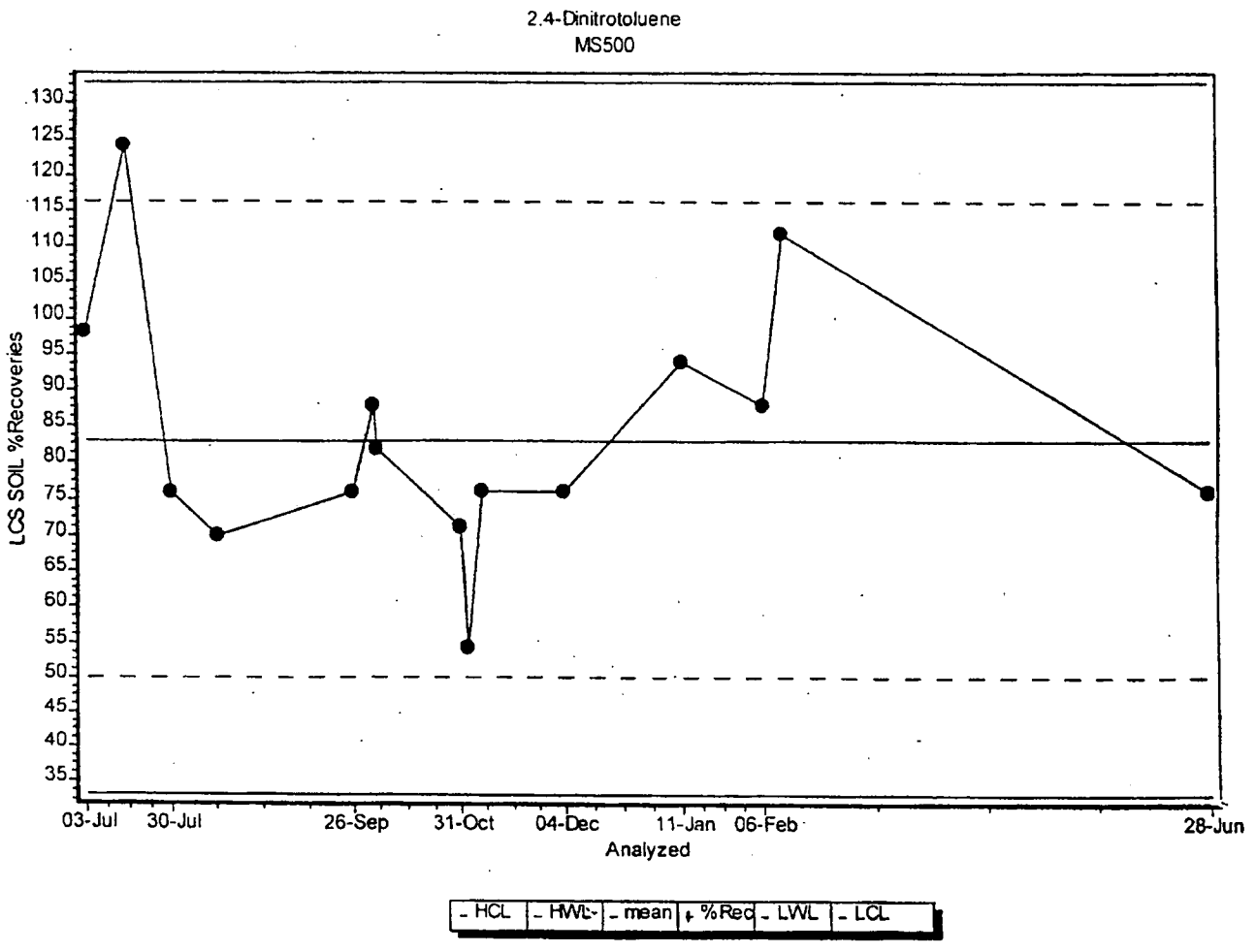
ATTACHMENT 3
ICP Metals Recoveries

ANALYST--->												
ANALYZED-->		2002-01-02	2002-01-03	2002-01-03	2002-01-04	2002-01-04	2002-01-11	2002-01-17	2002-01-18	2002-01-22	2002-01-22	2002-01-22
INSTRUMENT->		TJA ET1	TJA ET1	TJA ET1	TJA ET1	TJA ET1	TJA ET1	TJA ET1	TJA ET1	TJA ET3	TJA ET1	TJA ET1
TEST->		MT915P	MT915P	MT915P	MT915P	MT915P	MT915P	MT915P	MT915P	MT915P	MT915P	MT915P
QASEQ---->		011220TI3	011220TI2	011220TI2	011228TI1	020103TI1	020111TI1	020116TI1	020118TI1	020121IT8	020121IT6	020121IT7
MATRIX---->		W	W	W	W	W	W	W	W	W	W	W
LABID---->		LCW011220	LCW011220	LCW011220	LCW011228	LCW020103	LCW020111	LCW020116	LCW020118	LCW020121	LCW020121	LCW020121
REF----->		749835	750078	750109	750823	750836	751669	753092	753828	754470	754886	754902
Parameter	Units											
Aluminum	ppb		91.8	91.9	98.6	94.6	92.6	96.9	93.0	104.7	96.4	
Antimony	ppb	102.8	97.5	98.4	101.9	99.8	103.7	108.8	104.7	107.3	107.1	103.2
Arsenic	ppb		99.6	98.8	108.5	103.6	99.0	106.1	102.0	106.6	103.5	
Barium	ppb		96.3	96.6	102.8	103.9	96.9	101.5	97.3	102.1	100.2	
Beryllium	ppb		104.3	104.0	107.4	109.0	106.4	113.1	109.1	105.3	109.7	
Boron	ppb		101.9	101.8								
Cadmium	ppb		98.4	98.1	107.3	101.4	98.9	105.6	100.4	101.7	103.3	
Calcium	ppb		98.1	98.4	106.9	105.4	97.0	103.1	97.2	102.2	100.3	
Chromium	ppb		98.2	97.4	105.7		99.0	103.5	98.6	103.8	100.6	
Cobalt	ppb		98.0	96.7	104.1	99.5		100.9		101.4	98.7	
Copper	ppb			96.3	101.2	99.5			97.8	106.7	103.3	
Iron	ppb		102.5	103.0	109.7	103.6	104.1	106.0	105.8	102.2	109.2	
Lead	ppb			98.3	105.2	100.9	97.5	101.4	97.3	104.8	99.5	
Magnesium	ppb		98.8	96.5				101.7	97.3	104.0	97.8	
Manganese	ppb		99.1	98.6	106.5	101.7		103.8	99.7	102.6	101.0	
Molybdenum	ppb		97.5	97.8	102.3	101.4	97.2	103.4	98.2	102.4	99.8	
Nickel	ppb											
Potassium	ppb									92.1		
Selenium	ppb		103.6	103.9	112.1	108.7	103.9	112.0	107.6	109.3	110.8	
Silver	ppb	88.9	94.6	92.9	101.7	94.8	95.8	95.0	92.5	103.8		96.2
Sodium	ppb									109.9		
Strontium	ppb		98.2	97.8	104.0	105.0	97.0	101.9	97.4		100.4	
Thallium	ppb		101.3	101.2			100.9		104.2	108.6		
Tin	ppb		97.7	96.2	103.0	99.7	97.7	100.1	94.4		99.6	
Titanium	ppb		97.9	97.4	104.3	102.7	97.3	102.6	97.7	102.9	100.6	
Vanadium	ppb		98.4	97.5	105.4	100.8	99.0	103.4	99.6	102.4	101.1	

ATTACHMENT 4
Copper Recoveries



ATTACHMENT 5
2,4-Dinitrotoluene Recoveries

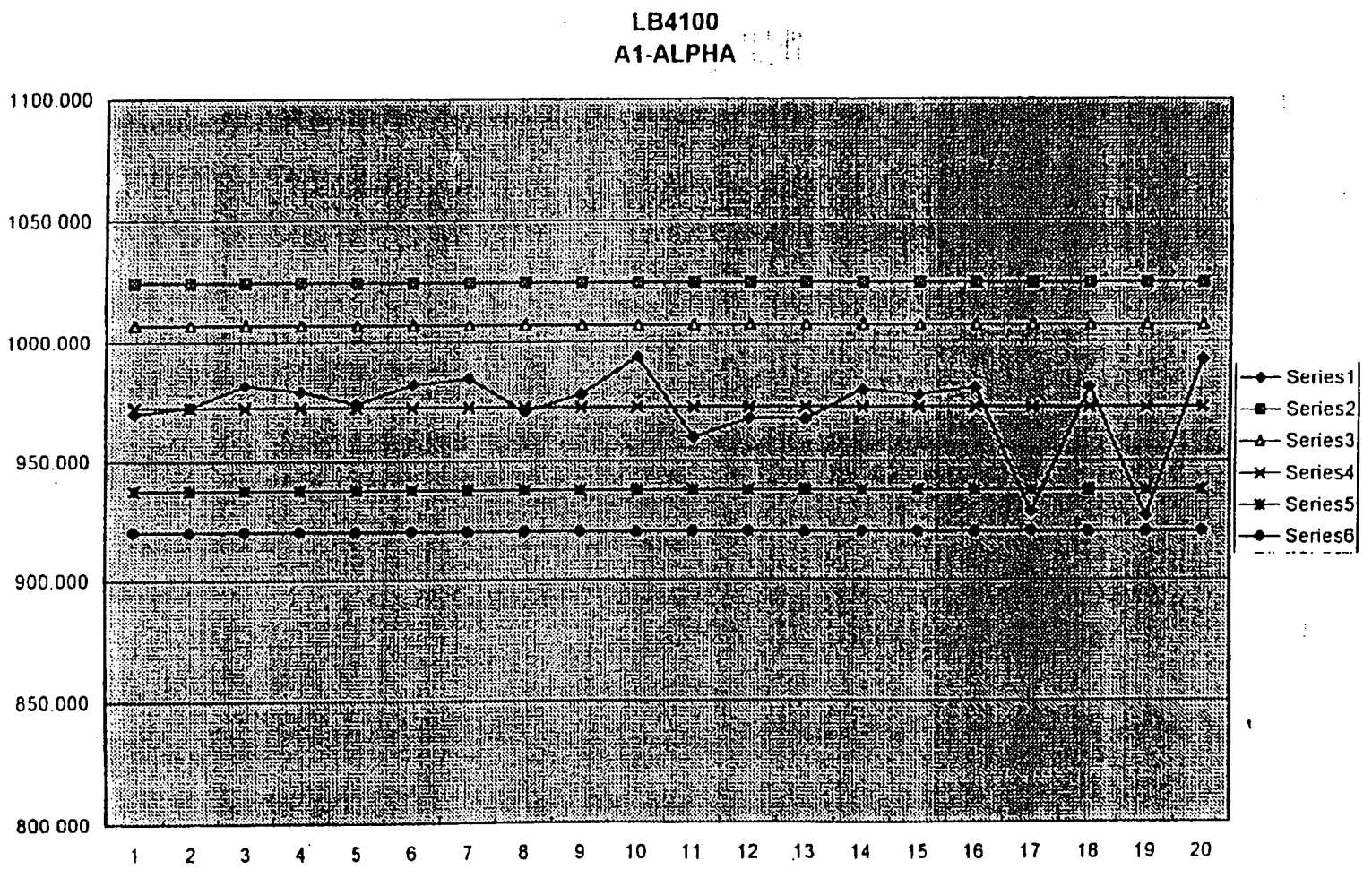


ATTACHMENT 6
Radiochemical Tolerance Charts: LB-4000 A/B Source Check

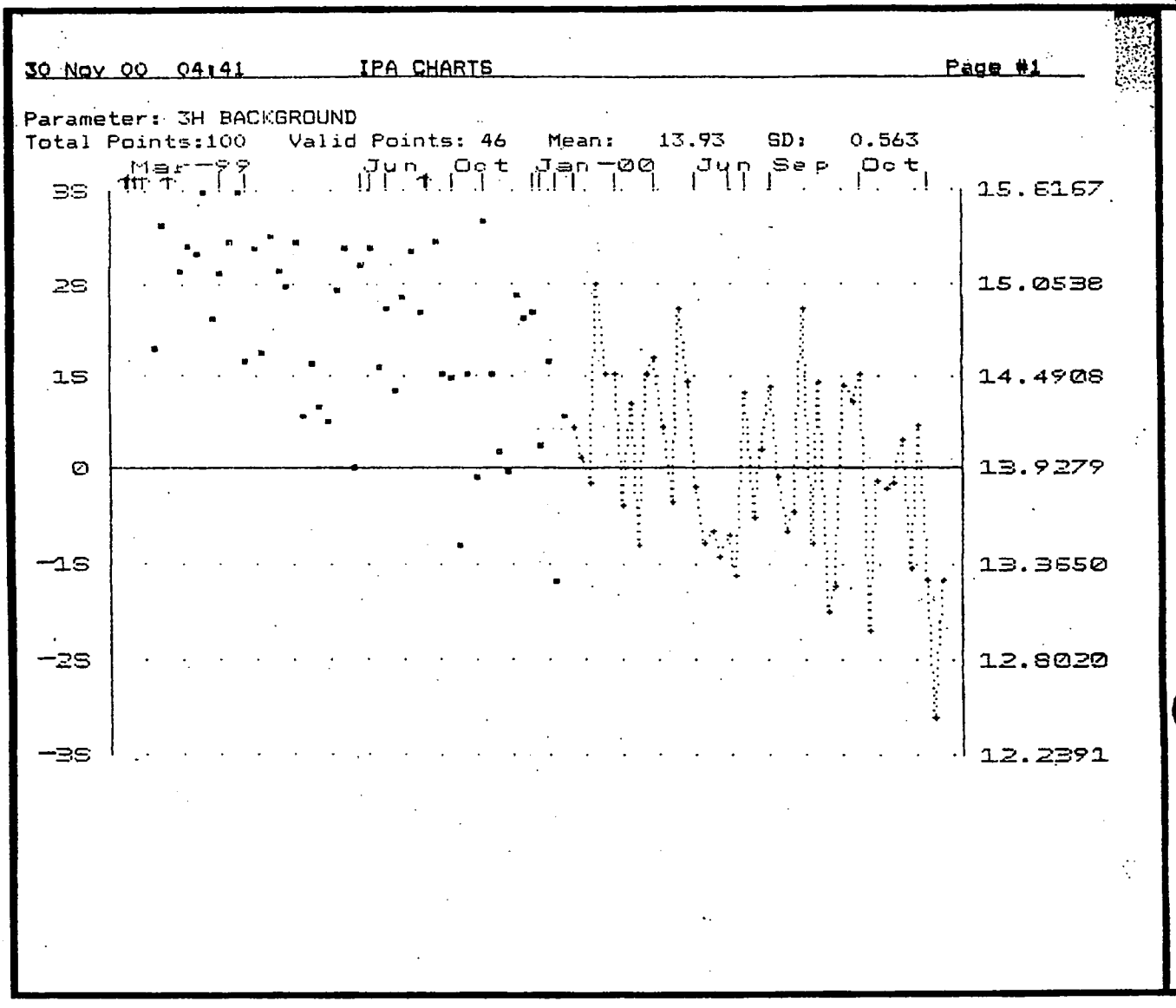
LB-4000 A/B SOURCE CHECK
CONTROL LIMITS

POINT	GPC SRC DATE	ALPHA SOURCE CPM A1	A2	A3	A4	ALPHA SOURCE CPM B1	B2	B3	B4
	Paste below:								
1	**08/10/2000**	975.730	871.070	999.670	1011.270	1222.400	2034.870	1007.730	974.870
2	**08/14/2000**	987.070	886.800	1005.870	1017.600	1238.530	2062.730	1009.930	962.330
3	**08/15/2000**	981.400	874.130	1005.730	1019.530	1239.730	2059.470	1010.600	952.670
4	**08/16/2000**	974.670	886.130	1008.670	1008.930	1227.200	2039.330	1011.200	935.530
5	**08/17/2000**	966.130	876.270	1004.670	1030.400	1235.070	2065.600	1011.730	940.870
6	**08/18/2000**	994.000	884.870	1001.470	1022.270	1224.000	2018.670	1014.730	955.800
7	**08/21/2000**	986.130	890.670	988.930	1014.330	1237.130	2041.330	1003.530	963.870
8	**08/22/2000**	960.270	829.670	964.870	966.930	1220.870	2045.130	1002.470	954.800
9	**08/23/2000**	969.200	872.130	1005.270	1024.330	1244.000	2056.330	1002.270	950.870
10	**08/24/2000**	983.730	882.870	990.330	1022.530	1228.870	2039.670	1004.400	953.400
11	**08/25/2000**	983.130	878.470	988.670	1019.330	1258.070	2045.330	1011.000	973.400
12	**08/28/2000**	989.870	891.600	1001.470	1008.800	1251.600	2049.400	1008.530	948.800
13	**08/29/2000**	1001.270	892.930	985.930	1004.200	1226.870	2039.000	1003.930	962.130
14	**08/30/2000**	990.000	884.670	989.530	1022.800	1237.070	2025.470	1008.800	970.130
15	**08/31/2000**	988.070	887.600	1020.130	1014.930	1223.800	2045.800	1008.070	970.600
16									
17									
18									
19									
20									
	MEAN	982.04	879.33	997.41	1013.88	1234.35	2044.54	1007.93	958.00
	UL@ 3 STDV	***1024.51***	***920.91***	***1064.06***	***1063.47***	***1283.57***	***2233.30***	***1087.54***	***1023.22***
	UW@ 2 STDV	*1007.09*	*904.93*	*1039.68*	*1044.07*	*1262.12*	*2164.18*	*1057.78*	*996.00*
	LW@ 2 STDV	*937.44*	*841.00*	*942.15*	*966.47*	*1176.35*	*1887.73*	*938.73*	*887.14*
	LL@ 3 STDV	***920.03***	***825.02***	***917.77***	***947.07***	***1154.90***	***1818.61***	***908.97***	***859.92***

ATTACHMENT 7
Radiochemical Tolerance Charts: LB4100 A1-Alpha



ATTACHMENT 8
Radiochemical Tolerance (IPA) Charts



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

TM

Standard Operating Procedure

Definition & Determination of Detection Limits

Document No.: SWL-GA-113

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APPROVALS	DATES
Procedure Prepared By	11/15/02 Date
Laboratory Manager or Director	11/04/02 Date
Laboratory Safety Officer	10/18/02 Date
Laboratory QA/QC Officer	10/18/02 Date

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

Document Status

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DEFINITION & DETERMINATION OF DETECTION LIMITS

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1.0 PURPOSE AND APPLICATION

1.1 The purpose of this document is to set forth the procedures for determining Instrument Detection Limits (IDLs), Method Detection Limits (MDLs), and Practical Quantitation Limits (PQLs). This procedure is designed for applicability to a wide variety of sample types.

1.2 The reporting Limit as presented on the Laboratory reports as "detection limit" is defined by the in house analytical SOP. This limit may correspond to the PQL, CRQL, CRDL or other limits as appropriate for the reference method, client or contract. The use of PQLs and other contract required limit determinations (CRDLs or CRQLs) in reporting results are determined first by any specific customer contract requirements. If there are no contract specific requirements, then the applicable reference method requirements for limit reporting will be observed, and finally if there are no method requirements for reporting, the reporting limits as defined in each SWL analytical SOP will be observed. For contracts/projects requiring the use of non-EPA approved methods, the laboratory shall establish detection limits according to the contract requirements and protocol.

1.3 All MDL's will be determined annually for each instrument, method, matrix and analyte. MDL studies will be performed if the section supervisor determines that a significant change in the analytical process has occurred.

1.4 Empirical verification checks will also be analyzed at approximately ½ MDL; MDL; and 2 x MDL. This is to be performed after the statistical MDL study and used for Sandia Projects.

1.5 MDL Check Samples (at approximately 2 times MDL) will be analyzed after the annual MDL study and on a quarterly basis.

2.0 RESPONSIBILITIES

2.1 It is the responsibility of the Organic and Inorganic Program Managers as well as the Section Supervisors to initiate the annual MDL studies for each method, matrix, instrument and analyte. It is the responsibility of the Inorganic Program Manager as well as the Section Supervisors to initiate the Quarterly IDLs for each metals method and analyte. It is the responsibility of the Organic and Inorganic Program Managers as well as the Section Supervisors to maintain all raw data and computations for the MDL and IDL studies. The raw data associated with each MDL including computations and spreadsheet printouts shall be maintained as part of the record archival and retrieval system for the test method analytical data.

2.2 The QA Department shall maintain and update the laboratory's compilation of all MDLs/PQLs as the SWL "Red Book". The Redbook is available electronically in each department.

3.0 EQUIPMENT

Refer to the listing in each specific Standard Operating Procedure for the required equipment.

4.0 PRECAUTIONS

Refer to the listing in each specific Standard Operating Procedure for the required equipment.

5.0 PROCEDURE

5.1 Southwest Laboratory's Instrument Detection Levels are based on the Statement of Work for the Environmental Protection Agency's Contract Laboratory Program (EPA CLP, SOW ILM 04.0).

IDL's are performed at the frequency required by the reference test method and using the procedure defined in the reference method. IDL's are performed quarterly for all CLP data using the latest SOW. In addition IDL's may be required for project specific, or client specific requests. IDL's are performed prior to performing client samples, unless the client has provided written authorization to depart from this policy. All client or project specific variations to performing IDL's will be in writing and retained in the client file records.

5.2 Method Detection Limit (MDL). Southwest Laboratory (SWL) uses the procedure outlined in "40 CFR part 136, Appendix B, Determination of Method Detection Limits".

5.2.1 The MDL for an analytical procedure may vary as a function of sample type. The procedure requires a complete, specific, and well defined analytical method. It is essential that all sample processing steps of the analytical method be included in the determination of the method detection limit.

5.2.1.1 The MDL obtained by this procedure is used to judge the significance of a single measurement of a future sample.

5.2.1.2 The MDL procedure was designed for applicability to a broad variety of physical and chemical methods. To accomplish this, the procedure was made device- or instrument-independent.

5.2.2 Procedure

5.2.2.1 Make an estimate of the detection limit using one of the following:

(a) The concentration value that corresponds to an instrument signal/noise ratio in the range of 2.5 to 5. If the criteria for qualitative identification of the analyte is based upon pattern recognition techniques, the last abundant signal necessary to achieve identification must be considered in making the estimate.

(b) The concentration value that corresponds to three times the standard deviation of replicate instrumental measurements for the analyte in reagent water.

(c) The concentration value that corresponds to the region of the standard curve where there is a significant change in sensitivity at low analyte concentrations, i.e., a break in the slope of the standard curve.

(d) The concentration that corresponds to known instrumental limitations. It is recognized that the experience of the analyst is important to this process. However, the analyst must include the above considerations in the estimate of the detection limit.

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

5.2.2.3 If the MDL is to be determined in reagent water (blank), prepare a laboratory standard (analyte in reagent water) at a concentration which is at least equal to or in the same concentration range as the estimated MDL. (Recommend between 1 and 5 times the estimated MDL). Proceed to Step 5.2.2.4.

Soil matrix MDL determinations are performed by mixing the analytical standard for the analyte to be determined with a known amount of clean sand or teflon beads (metals) at a concentration which is at least equal to or in the same concentration range as the estimated MDL. (Recommend between 1 and 5 times the estimated MDL). Proceed to Step 5.2.2.4.

If the MDL is to be determined in another sample matrix, analyze the sample. If the measured level of the analyte is in the recommended range of one to five times the estimated MDL proceed to Step 5.2.2.4. If the measured concentration of analyte is less than the estimated MDL, add a known amount of analyte to bring the concentration of analyte to between one and five times the estimated MDL. If the measured level of analyte is greater than five times the estimated MDL, there are two options:

(a) Obtain another sample of lower level of analyte in same matrix if possible.

(b) The sample may be used as is for determining the MDL if the analyte level does not exceed 10 times the MDL of the analyte in reagent water. The variance of the analytical method changes as the analyte concentration increases from the MDL, hence the MDL determined under these circumstances may not truly reflect method variance at lower analyte concentrations.

5.2.2.4 Take a minimum of seven aliquots of the sample to be used to calculate the MDL and process each through the entire analytical method. If more than 7 aliquots are processed, exclusion of data should not be done unless supported by both statistical outlier tests and suspicions of method performance. Use all valid data points to support MDL calculations to avoid inducing a systematic bias. Make all computations according to the defined method with final results in the method reporting units. Method blank measurements are used to determine the level of analyte, introduced by the analytical system. The blank measurement is evaluated to determine whether any contamination introduced via the analytical system could affect the MDL study.

5.2.2.5 It may be economically and technically desirable to evaluate the estimated MDL before proceeding with 5.2.2.4. This will: (1) prevent repeating this entire procedure when the costs of analyses are high, and (2) ensure that the procedure is being conducted at the correct concentration. It is quite possible that an incorrect MDL can be calculated from data obtained at many times the real MDL even though the background concentration of analyte is less than five times the calculated MDL. To ensure that the estimate of the MDL is a good estimate, it is necessary to determine that a lower concentration of analyte will not result in a significantly lower MDL. Take two aliquots of the sample to be used to calculate the MDL and process each through the entire method, including blank measurements as described above in 5.2.2.4. Evaluate these data:

(1) If these measurements indicate the sample is in the desirable range for determining the MDL, take five additional aliquots and proceed. Use all seven measurements to calculate the MDL.

(2) If these measurements indicate the sample is not in the correct range, re-estimate the MDL, obtain new sample as in 5.2.2.3, and repeat either 5.2.2.4 or 5.2.2.5.

5.2.2.6 Calculate the variance (S^2) and standard deviation (S) of the replicate measurement and MDL as follows:

$$S^2 = \frac{1}{n-1}\left(\sum_{i=1}^{n} X_i^2 - \left(\sum_{i=1}^{n} X_i\right)^2 \Big/ n\right)$$

$$S = (S^2)^{1/2}$$

where:

Xi: i = 1 to n, are the analytical results in the final method reporting units obtained from the n sample aliquots and Σ refers to the sum of the X values from i = 1 to n.

a) Compute the MDL as follows:

$$MDL = t_{(n-1,\ 1-\alpha = 0.99)}\ (S)$$

where:

MDL: the method detection limit

$t_{(n-1,\ 1-\alpha = 0.99)}$: the students' t value appropriate for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom. See Table.

S: standard deviation of the replicate analyses

b) The 95% confidence interval estimates for the MDL derived in (a) are computed according to the following equations derived from percentiles of the chi square over degrees of freedom distribution (χ^2/df).

LCL: 0.64 MDL
UCL: 2.20 MDL

where:

LCL and UCL: are the lower and upper 95% confidence limits respectively based on seven aliquots.

Tables of Students' t Values at the 99 Percent Confidence Level

Number of Replicates	Degrees of Freedom (n-1)	$t_{(cn^{-1},.99)}$
7	6	3.143
8	7	2.998
9	8	2.896
10	9	2.821
11	10	2.764
16	15	2.602
21	20	2.528
26	25	2.485
31	30	2.457
61	60	2.390
00	00	2.326

5.2.2.7 Reporting — The analytical method used must be specifically identified by number or title and the MDL for each analyte expressed in the appropriate method reporting units. If the analytical method permits options, which affect the method detection limit, these conditions must be specified with the MDL value. The sample matrix used to determine the MDL must also be identified with MDL value. Report the mean analyte level with the MDL. If a laboratory standard or a sample that contained a known amount analyte was used for this determination, report the mean recovery, and indicate if the MDL determination was iterated.

If the laboratory standard or sample level of the analyte in the sample matrix was below the determined MDL value or exceeds 10 times the MDL of the analyte in reagent water, do not report a value for the MDL. Re-estimate and re-perform the MDL determination in this procedure.

The % recovery of the analyte is compared to individual analytical method requirements. Should recoveries be below method requirements or gross method blank contamination exist; then the MDL study should be repeated.

All MDL raw data, calculations and results must be approved by the QA Manager before the can be used by the laboratory. After approval by the QA officer, or their designee, the MDLs are recorded by the QA Department in "The Red Book" (SWL-RDBK) and can be used for reporting as outlined in each analytical SOP.

The reporting limit may be the method specified (i.e. the CRQL in CLP) or method defined practical quantitation limit as specified in the RCRA methods or other method defined sensitivity. In all cases the establishment of the reporting limit is documented in the in house analytical SOPs. The reporting limit must never be less than the calculated Method Detection Limit from the in-house method as determined by the laboratory.

5.2.2.8 To ensure the reasonable MDL values are determined, a MDL check sample (spiked at approximately 2 times MDL) is taken through the same process as the MDL. This is performed after the annual MDL study and then on a quarterly basis after that.

5.3 Practical Quantitation Limits (PQL) are also used in data assessment and reporting.

5.3.1 PQLs are defined as being a multiplicand of the laboratory MDL. PQLs are based on each of the reference methods and are defined for SWL in each in house analytical SOP. For USACE and NFESC projects the PQL is 3 to 5X MDL unless specified otherwise by the Projects QAPP.

5.3.2 For those projects or methods that do not specify PQLs, SWL uses the MDL seen for the parameter/matrix/method combinations as a basis for determining a PQL; i.e. water based MDLs produce water based PQLs and soil based MDLs produce soil based PQLs. In situations when uncommon materials are encountered, PQLs may be extrapolated from water based MDLs. Specifics for each analyte can be found in the SWL analytical SOPs.

5.3.3 The PQL is the level at which the low standard of the calibration curve is prepared. Data results detected between the MDL and PQL will be qualified as estimated (i.e. by a data qualifier "flag"). When sample results exceed the high standard in the calibration curve, the sample will be diluted and reanalyzed (if possible) to bring them within the calibration curve. Sample results that exceed the high standard will be flagged as such.

6.0 RECORDS

6.1 The raw data associated with each MDL including computations and spreadsheet printouts shall be maintained as part of the record archival and retrieval system for the test method analytical data.

6.2 A compilation of MDLs/PQLs are maintained in the Laboratory's "Red Book" SWL GENRDBK. The "Red Book" is on file in the QA Department. Copies are in place in each department.

7.0 REFERENCES

- 40 CFR, Part 136, Appendix B.
- Glaser, J.A., Foerst, D.L., McKee, G.C., Quave, S.A., and Budde, W.L., "Trace Analysis for Wastewaters", Environmental Science and Technology 15, 1426 (1981).
- U.S. Environmental Protection Agency's Contract Laboratory Program Statement of Work ILMO4.O

8.0 ATTACHMENTS

Not Applicable.

9.0 DEFINITIONS

9.1 Instrument Detection Limits (IDL).

It is defined as the smallest signal above background noise that an instrument can detect reliably. It does not address possible blank contaminants or matrix interferences.

IDL for each analyte in a given method is determined for Inorganic ICP, CV (Hg) and Cyanide analyses. IDL's are determined by analyzing a standard (at a concentration of

3X to 5X the IDL) as a sample seven (7) times on three (3) non-consecutive days. The three standard deviations from each day are averaged and then multiplied by three(3) to obtain the IDL.

The IDL is only performed when required, such as for SOW ILM04.0.

9.2 Method Detection Limits (MDL).

The Method Detection Limit (MDL) is defined as the minimum concentration of a substance that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.

9.3 Practical Quantitation Limits (PQL)

The MDLs defined above produce detection levels that are not typically achievable in the analysis of real samples. To address this reality, numerous somewhat higher levels have been proposed and justified. These more realistic assessments of levels that can be reported have been called by many names (Real Levels, Reporting Levels, Contract Required Levels of Detection, etc.). SWL has chosen to use the term Practical Quantitation Limits (PQLs), since this seems to be in common use.

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GA-115

The Standard Operating Procedures (SOPs) contained herein are adopted by Pace Analytical Services.

These SOPs will be followed until superceded by Pace Analytical Services SOPs.

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

TM

Standard Operating Procedure

Sample Custodian

Document No.: SWL-GA-115

Rev. No. / Date: Rev. 9.0 — 09/12/03

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APPROVALS	DATES
Melissa Cranick Procedure Prepared By	9-12-03 Date
Program Manager or Section Supervisor	9-12-03 Date
Chuck Hoover Laboratory Safety Officer	9/12/03 Date
Chuck Hoover Laboratory QA/QC Officer	9/12/03 Date

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

Document Status

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SAMPLE CUSTODIAN

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1.0 PURPOSE & APPLICATION

The Sample Custodian is the person who initiates the receipt of samples into the laboratory. It is the purpose of this Standard Operating Procedure to make uniform the duties/procedures needed to ensure that sample integrity and documents associated with that integrity are properly performed.

2.0 RESPONSIBILITIES

The Sample Custodian (SC) for the laboratory has duties and responsibilities that include but are not limited to:

2.1 Receiving/Inspecting shipping coolers.

2.2 Noting the integrity/ temperature of samples on appropriate forms.

2.3 Signing Chain-of-Custody (COC) documents (Attachment 1).

2.4 Verifying agreement between the COC and the samples.

2.5 Taking pH of all applicable containers to verify sample preservation.

2.6 Logging samples; labeling containers.

2.7 Placing samples in appropriate storage.

2.8 Documenting cooler/ sample information on Cooler Receipt/Sample Log-in Sheet (Attachment 2).

NOTE: EPA has a separate log-in form: FORM-DC-1.

2.9 Distribution of paperwork to appropriate laboratories.

2.10 Archiving reported samples to appropriate storage.

2.11 Disposal of expired samples.

2.12 Calibration of temperature reading device.

2.13 Monitoring storage temperatures.

2.14 Maintaining a secure area.

3.0 EQUIPMENT

3.1 Box knife or equivalent.

3.2 Infra-red temperature Gun Raytech Rayngor Model ST-2 or equivalent.

3.3 pH paper, ranges: 1 to 6 pH and 12 to 14 pH.

3.4 Sample Transfer Carts.

3.5 LIMS Access.

3.6 HP Laser Jet Printer or equivalent.

4.0 PRECAUTIONS

Refer to the "*Laboratory Safety Plan*" SWL-GA-111 for safety precautions.

5.0 PROCEDURE

5.1 Sample Receipt

5.1.1 Receiving & Inspecting Samples.

5.1.1.1 Upon arrival at the Laboratory, all coolers will be accounted for via the FedEx® Package Tracking Summary (see Attachment 15). Summary Sheets will be maintained in Sample Receiving.

5.1.1.2 All sample-shipping coolers shall be identified as "potentially radioactive" or "non-radioactive." This process involves communicating with the project officer as to the "radioactive" status of said sample shipping coolers. Therefore, the Project officers shall notify the Sample Custodian of expected "radioactive" samples prior to sample receipt. In some cases, the Project Officer will not know that "radioactive" samples are expected. In this case, the Project Officer, after consultation with the laboratory Radiation Safety Officer (RSO), shall notify the Sample Custodian of which clients send "radioactive" samples for processing. If sample coolers arrive from unknown origins, then the RSO shall be notified and consulted before proceeding.

Potentially radioactive coolers shall only be handled by the RSO or designee, and inspected for the following and documented on the Cooler Receipt/Sample Log-In Sheet (see Attachment 2). Details on the radioactive screening process are found in SOP SWL-RD-139, "*Radioactive Sample Screening*."

- Condition of cooler
- Presence/absence, and condition of tamper-evident Custody Seals. When Custody Seals are not tamper-evident a notation of "Not Tamper-evident" will be made under "Custody Seals": on the Cooler Receipt/Sample Login Sheet.
- Labeling on cooler

5.1.1.3 Coolers are identified on the Cooler Receipt/Sample Log-In Sheet as follows:

- Client code
- Date
- Container # (Example: EPA-09/01/97-1)

5.1.2 After hour receipt

5.1.2.1 For after-hour sample receipt, a designated person shall receive the samples and store them properly for sample log-in processing the next business day.

5.1.2.2 The designee shall leave a notice in Sample Receiving showing what was received and the refrigerator number in which the cooler is located.

5.1.3 Opening Cooler

5.1.3.1 All shipping containers shall be opened under an exhaust hood or an approved, well-ventilated area.

5.1.3.2 The temperature of a representative sample or provided temperature blank is measured using the non-contact I.R thermometer gun or (when qualified by client) by submerging an immersion thermometer into the temperature blank bottle. To arrive at a representative sample the SC will measure the temperature of two samples from the cooler. If the two sample temperatures agree, this temperature is used. If the two are in disagreement, a third sample is measured and if that measurement is in agreement with one of the other two, that temperature will be used as the representative temperature. If all three measurements are in disagreement, the three temperatures will be communicated to the client (via the Project Officer) for resolution.

5.1.3.3 The COC is removed from the cooler, and the temperature (corrected for bias) is recorded in the lower right corner and on the Cooler Receipt/Sample Log In Sheet (see Attachment 2). Temperature excursions will also be documented with a "P.O. Notified" check box. PO's are to submit client notification/resolution documentation for inclusion with the paperwork. The bias of the thermometers from the daily calibration check (see section 5.8) is determined and recorded in the Zone Book (see Standard Operating Procedure "*Maintenance of ZONE Log Books*," SWL-GA-128). Corrective Action is required when the bias exceeds ±2°C.

5.1.3.4 The COC is signed by the Sample Custodian.

5.1.3.5 A copy of the COC and all applicable sample receipt documents is/are taken to the PO for completion of an Analytical Request Form (AR

The PO will review the COC and communicate to the client any cooler temperature excursions and any unclear technical directions that might be recorded. Resolution on how to proceed will be documented and attached to the paperwork.

An ARF (see Attachment 3) consists of the following:

- Client info./client code
- Project name/number
- Sample ID from COC
- Sample matrix
- Analysis to be performed
- Turn around time
- Special Provisions
- Deliverable information

5.2 Sample Log-In

5.2.1 Sample Verification

5.2.1.1 Samples are removed from the cooler and set out in the order of the ARF form. At this time any problems with the samples are noted on the Cooler Receipt/Sample Login Sheet. Any problems discovered may consist of the following:

- Breakage
- Leakage
- Sample ID discrepancy on the COC verses the sample bottle and vice versa.
- Incorrect preservative or absence of preservative (Attachment 13).
- Insufficient volume for analysis requested.
- Odor of a sample.
- Presence of headspace in volatile samples.

5.2.1.2 The SC verifies pH of all applicable containers for sample preservation, including Residual Chlorine Checks (see Attachment 13). Verification procedures are as follows:

- Disposable plastic cups are laid out corresponding to the layout of samples on table.

- A small piece of pH or starch Iodine paper is placed in the bottom of each plastic cup.

NOTE: The pH must be less than 2, for 1-6 pH paper used for acids. The pH must be greater than or equal to 12, for 12-14 pH paper used for caustic preservation. For residual chlorine checks, the starch Iodine paper should show no color change.

- The SC pours a small amount of the sample into its plastic cup, watching the paper for a change in color (which determines the pH level).
- The SC shall document level of pH on the Cooler Receipt/Sample Log-In Sheet and notify the PO if any of the following are also determined:

Absence of a preservative
Incorrect preservative present
pH above or below required range
Residual Chlorine detected in applicable samples
Incorrect analysis and/or preservative labeling

The PO may then decide to add preservation in the laboratory.

Documentation (including before and after readings, time, date of adjustment) is noted on the Sample Log In form (comments section.) A client/PO email or telephone log is attached.

For metals preservation only, Trace Metals grade HNO_3 is used. Lot numbers are reported on the Sample Log In form. The pH is again checked and recorded, and then checked and recorded again after 16 hours equilibrium period. Radioactive chemistry samples require a 24-hour equilibrium time. Time and date of all adjustment readings are documented.

5.2.2 Procedure for Log-In of Samples

5.2.2.1 After samples are set out and all pertinent information has been recorded, the samples are then logged into the Laboratory Information Management System (LIMS).

- A group of samples is assigned a unique number called an episode number. This number is generated by the LIMS system and is a sequential, non-repeating number. (e.g. 23456).
- Each sample in the episode is assigned a unique sample number. Sample numbers follow the episode number and a decimal point. (e.g. 23456.01, 23456.02).
- Individual sample containers are assigned a unique alpha character to assure each container received is accounted for in the LIMS. (e.g. 23456.01A, 23456.01B, 23456.02A, 23456.02B)

5.2.2.2 As samples are logged in, LIMS automatically generates a worksheet (Attachment 4) and container labels simultaneously.

- The worksheet gives a full scope of the samples logged in, identifying each sample separately and the specific analysis for each sample bottle.
- The LIMS system is capable of assigning 99 samples per episode, and 26 containers per sample number.

5.2.2.3 Following a login review (see "*Sample Login Review*" SWL-GA-135), photocopies of the worksheet, along with any associated paperwork, are distributed to the appropriate laboratory departments. This worksheet package consists of the following:

- LIMS worksheet
- Any special compound lists and/or attachments provided by the client or PO
- COC documents (signed/dated by Sample Custodian)
- Cooler Receipt / Sample Log-In Sheet.
- ARF
- Shipping document/ Airbill

The SC retains an additional copy of the worksheet package for the Sample Receiving area and sends the original to the Reporting Department files.

5.3 Sample Storage

5.3.1 Immediately after samples have been logged in and labeled with laboratory ID, they are put into their appropriate storage area.

5.3.2 All refrigerators in the Sample Receiving area are designated for specific types of samples. (See Attachment 14). Listed below are the refrigerators currently located in the Sample Receiving area and Radioactive Sample Receiving Area:

5.3.2.1 #7 Volatile Samples

5.3.2.2 #4 EPA volatile samples

5.3.2.3 #8 Inside walk-in

5.3.2.4 #9 Current walk-in

5.3.2.5 #12 Archive walk-in

5.3.2.6 #18 Radioactive Materials Controlled Area (RMCA archive walk-in)

5.3.2.7 #R-1 Rad Samples

5.3.2.8 #R-2 Rad Volatile Samples

5.3.3 Descriptions of Appropriate Storage for Each Refrigerator

5.3.3.1 The #7 refrigerator contains only volatile samples - both water and soil matrices.

5.3.3.2 The #4 EPA refrigerator contains only EPA volatile samples.

5.3.3.3 The #8 walk-in contains archived volatile samples, as well as samples to be transferred to other facilities for outside service.

5.3.3.4 The #9 walk-in contains all in-use (current) organic and inorganic samples. No potentially radioactive samples or volatiles samples are stored in #9.

5.3.3.5 Walk-in #12 contains samples that are already reported and have been archived into storage, awaiting disposal in 90 days.

5.3.3.6 Aqueous metal samples for Navy projects will be stored on shelves in the sample receiving area (stored at room temperature), unless specified otherwise by the client's QAPP.

5.3.3.7 The RMCA Sample Receiving Area (#R-1) sample refrigerator restricted to non-volatile potentially radioactive samples.

5.3.3.8 The RMCA Sample Receiving Area (#R-2) volatile sample refrigerated is restricted to potentially radioactive volatile samples.

5.3.3.9 The RMCA Archive walk-in (#18) contains and is restricted to potentially radioactive samples. Authorized personnel only are allowed in this walk-in!!!

5.3.4 Southwest Laboratory of Oklahoma is a secure facility, allowing access by authorized personnel only to the storage facilities.

5.3.5 Internal Chain-Of-Custody. An Internal (COC) is generated from LIMS at the same time as samples are put into storage. The ICOC (Attachment 8) is put in a 3 ring binder outside the sample storage units; analysts are to sign samples out and into storage via use of this form. Samples that are used and not returned to storage are logged into the depleted bottle log. (Attachment 11).

5.4 Sample Document Verification

5.4.1 Verification of Documents

5.4.1.1 The SC will compare the following documents to verify agreement among the information contained on them:

- COC
- Sample tags
- Analytical Request Form

- The SC shall document agreement and note any discrepancies found among the forms on the Cooler Receipt/Sample Login sheet.

5.4.1.2 The SC will note and record problems in the "remarks" box.

- SC may reference another form, such as the COC, that details the problems.
- The SC reports discrepancies to the PO for client clarification. The discussion and resolution are documented on the Client/Laboratory Communication Form (Attachment 5). These forms are then included in the Case File.

5.5 Sample Splitting/ Facility Transfer

5.5.1 Procedure for Splitting Samples

When clients supply their own containers or when bulk samples are received, the SC may split the samples to provide sufficient aliquots for each analysis to be performed. To determine the manner in which samples are split, the PO will notify the client of the necessity to split the sample and request his instructions.

5.5.2 Procedure for Facility Transfer

5.5.2.1 For samples which require analytical protocols performed in one of SWLO affiliated laboratories or an outside laboratory, a Facility Transfer Sheet or Outside Analytical Services Request Form are completed (Attachment 6 & 7).

5.5.2.2 The form is sent to that facility along with the samples and a copy of the worksheet, and client Chain-of-Custody forms.

5.6 Log-in Modification/Cancellation

5.6.1 A Sample Modification/Cancellation Request form (Attachment 9) should be completed and approved for any modification or cancellation requests of a sample or analysis.

5.6.2 Upon receipt of this Modification Request Form, the SC shall update LIMS accordingly.

5.6.3 When modification or cancellation is completed in LIMS, the original form will accompany a corrected worksheet to the project file. A copy of the new worksheet will be kept in Sample Receiving, and additional copies will be distributed to the laboratory.

5.7 Sample Archive

5.7.1 Each morning the LIMS generates a list of samples ready to be archived. These selected samples were reported the previous day. The archive list (Attachment 12) shows the SC the following:

- SWLO sample ID
- Client code
- Client sample ID
- Matrix
- Original log-in date
- Archive date
- Disposal date

5.7.2 The SC uses the archive list to record the moving of spent samples from walk-in #9 to walk-in #12 for an approximate 90-day storage.

5.7.2.1 Samples found on the archive list are taken off the shelf in #9 and placed into a box, as the SC fills out an inventory list for the box.

5.7.2.2 Once a box is full, the SC tapes the inventory list to the box. This allows the SC to determine the contents of the box.

5.7.2.3 Soils and waters are archived separately to eliminate sorting them out at the time of disposal.

5.7.3 When pulling archival boxes from walk-in #12 for disposal, the SC uses the disposal date on the archive list to determine which samples have expired. Disposal procedures may be found in "*Hazardous Waste Management Plan*", SWL-GA-114).

5.8 Calibration Check of thermometers

5.8.1 The thermometer(s) is/are checked against one of the NIST traceable sample receiving thermometers.

5.8.1.1 The check is performed daily.

5.8.1.2 The check is documented in the Zone Book with the thermometer reading, the NIST traceable thermometer reading, and the NIST traceable thermometer I.D. #.

5.9 Monitoring Storage Temperatures.

5.9.1 The SC is responsible for monitoring the temperature of all storage refrigerators in the Sample Receiving area each morning, excluding weekends and holidays.

5.9.2 A glycerin thermometer including a serial number can be found in each refrigerator.

5.9.3 The temperature [corrected for bias (section 5.8)] from each thermometer is recorded in the SR Zone Calibration Checks log found in the Sample Receiving area. This log provides a specific entry space for each refrigerator and for every day of the month. (See Attachment 10), and allows the SC to record:

5.9.3.1 Time of the check.

5.9.3.2 His/ Her Initials.

5.9.3.3 Temperature, to the nearest 0.5°.

5.9.3.4 Affirmation that the temperature recorded was within accepted limits.

5.9.3.5 Corrective Action.

- If the temperature is outside of accepted limits, the laboratory manager or designee shall be notified immediately.
- In the event of unit failure, a repairman will be called out as soon as possible. The SC shall transfer all current samples to an alternate storage refrigerator temporarily.

6.0 RECORDS

6.1 Original field Chain of Custody, Airbills, Sample Tags, Cooler Receipt/Sample Log-In Sheet. The Project Officer's Analytical Request Form, and Laboratory work sheet go to data reporting's case files.

6.2 Copies of the above documents are maintained in a sequential three ring binder.

6.3 The Internal Chain Of Custody upon completion is also placed in the case file. (Maintained in the data reporting section).

6.4 Facility Transfer sheet to and from our Baton Rouge & St. Louis Facilities as with the Internal COC are also placed in the case file.

6.5 Zone Log records (see SWL-GA-128 "*Maintenance of ZONE Log Books*").

6.6 Depleted bottle log (see Attachment 11).

6.7 Sample Disposition Records (see SWL-GA-114 "*Hazardous Waste Management Plan*").

6.8 FedEx® Package Tracking Summary (see Attachment 15).

7.0 REFERENCES

Not Applicable.

8.0 DEFINITIONS

COC: .....Chain of Custody. Field document used here to document and relinquish custody of sample from field to Laboratory.

Internal COC:.Internal Chain of Custody. Document used to record custody of samples through the laboratory.

DC-1:.....Document Control-Form #1. Used as initial Laboratory sample log-in document.

ARF:.....Analytical Request Form. A form completed by the Project Officer and used by the SC, to detail what analyses is required for each sample.

LIMS:.....Laboratory Information Management System

RMCA:.....Radioactive Materials Controlled Area

9.0 ATTACHMENTS

See Attachment's 1 through 15

ATTACHMENT 1
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.
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TM

SAMPLING FIRM | CLIENT CONTACT | PHONE NUMBER

PROJECT NUMBER | PROJECT NAME

SAMPLERS: (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 LABORABORY BY: (Signature)
RELINQUISHED BY: (Signature)	DATE	TIME	RECEIVED BY: (Signature)	REMARKS:			

[SR012-0492-02]

Typist: LLR

ATTACHMENT 2a
Cooler Receipt / Sample Log-In Sheet
(Standard SWL Form)

COOLER RECEIPT / SAMPLE LOG-IN SHEET

COOLER RECEIPT / SAMPLE LOG-IN SHEET (115-ATT2.WB1) / SWL-GA-115 REV 5.0 / GA-115-CRLOGIN-F

LAB NAME: SOUTHWEST LABORATORY OF OKLAHOMA

PAGE ____ OF ____

RECEIVED BY (PRINT NAME): _____ REC'D DATE _____

RECEIVED BY (SIGNATURE): _____ TIME REC'D _____

LOGGED IN BY (SIGNATURE): _____ LOG-IN DATE _____

PROJECT:

EPISODE:

SAMPLE DELIVERY GROUP:

Remarks

1. CUSTODY SEAL(S): Present/Absent
   Intact/ Broken
2. CUSTODY SEALS NOS.:
   _____
   _____
3. CHAIN-OF CUSTODY. Present/Absent
   Sealed In Plastic? Yes/ No
   Taped To Lid? Yes/ No
   Properly Filled Out
   (Ink, Signed, ETC.)? Yes/ No
4. AIRBILL AirBil/ Sticker
   Present/Absent
5. AIRBILL NO:
   _____
   _____
6. COOLER CONDITIONS
   Enough Ice? Yes/ No
   Type of Ice? _____
   Type of Packing? _____
7. SAMPLE TAGS Absent
8. SAMPLE CONDITION:
   Intact/ Present/Broken*/ Leaking
   Bottles Sealed In Separate Plastic Bags? Yes/ No
   Correct Containers Used For Tests Indicated? Yes/ No
   Correct Preservative? Yes/ No
   Sufficient Sample? Yes/ No
   Labels Complete (I.D., Date, Time, Signature, Preservative? Yes/ No
   VOA Samples Without Bubbles? Yes/ No~
9. Does Information on Custody Records,Labels,Tags Agree? Yes/ No*
10. RAD SCREEN WITH GIEGER COUNTER? Yes/ No
11. P.O. Called? Yes/ No

Client Sample #	Sample Fraction @	Assigned LAB#	Cooler I.D.	pH Check	ACID/ BASE LOT#	REMARKS: CONDITION OF SAMPLE SHIPMENT, ETC.

* Contact PO and attach record of resolution
@ Sample Fractions: B=SV GC/MS, V= VOA GC/MS or GC, P=Pesticide,H=Herbicide, D=Dioxin, A=Air, I=Inorganics, C=Cyanide, M=Metals, R=Radiochemistry
~ Note samples with bubbles under remarks section.

ATTACHMENT 2b
EPA Cooler Receipt / Sample Log-In Sheet

SAMPLE LOG-IN SHEET

Lab Name SOUTHWEST LABORATORY OF OKLAHOMA	Page ____ of ____
Received by (Print Name)	Log-In Date
Received by (Signature)	
Case Number	Sample Delivery Group No. / SAS Number

Remarks:

1. Custody Seals(s)	Present/Absent* Intact/ Broken
2. Custody Seal Nos.	_____
3. Chain-of Custody Records	Present/Absent*
4. Traffic Reports or Packing Lists	Present/Absent*
5. Airbill	Airbill/ Sticker Present/Absent*
6. Airbill No.	_____
7. Sample Tags	Present/Absent*
Sample Tag Numbers	Listed/Not Listed on Chain-of-Custody
8. Sample Condition	Intact/ Broken*/Leaking
Cooler Temperature Indicator Bottle	Present/Absent*
9. Cooler Temperature	_____
10. Does Information on Custody records, traffic reports, and sample tags agree?	Yes/ No*
11. Date Received at Lab	_____
12. Time Received	_____

Sample Transfer	
Fraction	Fraction
Area #	Area #
By	By
On	On

EPA Sample #	Corresponding Sample Tag #	Corresponding Assigned Lab #	Remarks: Condition of Sample Shipment: etc.

* Contact SMO and attach record of resolution.

Reviewed By	Logbook No.
Date	Logbook Page No.

FORM-DC1 OLM04.2

ATTACHMENT 2c
Cooler Receipt / Sample Log-In Sheet
(Special Form used for Sandia)

COOLER RECEIPT / SAMPLE LOG-IN SHEET

COOLER RECEIPT / SAMPLE LOG-IN SHEET (115-ATT2.WB1) / SWL-GA-115 REV 5.0 / GA-115-CRLOGIN-F

LAB NAME: SOUTHWEST LABORATORY OF OKLAHOMA

PAGE ___ OF ___

RECEIVED BY (PRINT NAME): _____ REC'D DATE _____

RECEIVED BY (SIGNATURE): _____ TIME REC'D _____

LOGGED IN BY (SIGNATURE): _____ LOG-IN DATE _____

PROJECT:

EPISODE:

SAMPLE DELIVERY GROUP:

Remarks Cooler:

1. CUSTODY SEAL(S): Present/Absent
   Intact/ Broken
2. CUSTODY SEALS NOS.:
   _____
   _____

Custody Seals on Container Present/Absent
Intact/ Broken

3. CHAIN-OF CUSTODY. Present/Absent
   Sealed In Plastic? Yes/ No
   Taped To Lid? Yes/ No
   Properly Filled Out
   (Ink, Signed, ETC.)? Yes/ No
4. AIRBILL AirBill/ Sticker Present/Absent
5. AIRBILL NO:
   _____
   _____
6. COOLER CONDITIONS
   Enough Ice (Temp 2-6 deg C)? Yes/ No
   Type of Ice? _____
   Type of Packing? _____
7. SAMPLE TAGS Present/Absent
8. SAMPLE CONDITION: Intact/ Broken*/ Leaking
   Bottles Sealed In Separate Plastic Bags? Yes/ No
   Correct Containers Used For Tests Indicated? Yes/ No
   Correct Preservative? Yes/ No
   pH Checked on all samples Yes/ No
   except VOA's & Soils? Yes/ No
   Sufficient Sample? Yes/ No
   Labels Complete (I.D., Date, Time, Signature, Preservative? Yes/ No
   VOA Samples Without Bubbles? Yes/ No~
9. Does Information on Custody Records,Labels,Tags Agree? Yes/ No*
10. RAD SCREEN WITH GIEGER COUNTER? Yes/ No
11. P.O. Called? Yes/ No

Client Sample #	Sample Fraction @	Assigned LAB#	Matrix	pH Check	ACID/ BASE LOT#	Temp.	Has Notes

* Contact PO and attach record of resolution
@ Sample Fractions: B=SV GC/MS, V= VOA GC/MS or GC, P=Pesticide,H=Herbicide, D=Dioxin, A=Air, I=Inorganics, C=Cyanide, M=Metals, R=Radiochemistry
~ Note samples with bubbles under remarks section.

ATTACHMENT 3
Analytical Request Form

SOUTHWEST LABORATORY OF OKLAHOMA

ANALYTICAL REQUEST FORM, SWL-GA-131

Ref# 10220
Pg 1

Client LYONDELL CHEMICAL CO
Contact RON JENSEN
Project ARCO
Program
Location TURTLE BAYOU
SDG EPISODE Closed

Client Code: AATS/BR
Phone&Fax#: 281-862 5577 281-862 6148
PO Number:
Receipt Date 01/07/2003
Sample TAT: Fax Rpt 14

Reports
Data Pkgs
EDD ARCO

Special Provisions: TO-14 AND TBA
SEND REPORT TO AATS/BR

Client ID	Type	Matrix	MS499									
TB18415		A	X									
TB18416		A	X									

Reporting Address: LYONDELL CHEMICAL CO
Attn: Ron Jensen;SEVICE CENTER, #116,2502 SHELDON ROAD,CHANNELVIEW,TX 77530

Billing Address: Same As Reporting Address

Project Officer: JAYANT SHRINGARPURE

Prepared 01/07/2003

Typist: LLR

ATTACHMENT 4
Sample Log-In Record

Southwest Laboratory of Oklahoma
1700 W. Albany Suite C
Broken Arrow, OK 74012-1421

SAMPLE LOG-IN RECORD

REVIEWED
BY Scott Fitzgerald
DATE 12/20/00

Date 12/20/2000 Page1
Episode 45454
Client EPA
Project OLM04.2
Received 12/20/00
Logged In 12/20/00

TEST	PRI	DUE	CONTAINER	DESCRIPTION	EXTRACTED	ANALYST	ANALYZED
Sample: 45454.01		Sampled:12/19/00 07:45		ID:Y0318	SDG:45454 Matrix:	W	Containers: 5
MS320	4	01/09/2001	ABC	EPA - 25 ML			
MS519	4	01/09/2001	DE	BNA BY OLM04.2			
Sample: 45454.02		Sampled:12/19/00 09:10		ID:Y0319	SDG:45454 Matrix:	W	Containers: 5
MS320	4	01/09/2001	ABC	EPA - 25 ML			
MS519	4	01/09/2001	DE	BNA BY OLM04.2			
Sample: 45454.03		Sampled:12/19/00 09:15		ID:Y0320	SDG:45454 Matrix:	W	Containers: 5
MS320	4	01/09/2001	ABC	EPA - 25 ML			
MS519	4	01/09/2001	DE	BNA BY OLM04.2			
Sample: 45454.04		Sampled:12/19/00 09:25		ID:Y0321	SDG:45454 Matrix:	W	Containers: 5
MS320	4	01/09/2001	ABC	EPA - 25 ML			
MS519	4	01/09/2001	DE	BNA BY OLM04.2			
Sample: 45454.05		Sampled:12/19/00 09:55		ID:Y0322	SDG:45454 Matrix:	W	Containers: 5
MS320	4	01/09/2001	ABC	EPA - 25 ML			
MS519	4	01/09/2001	DE	BNA BY OLM04.2			
Sample: 45454.06		Sampled:12/19/00 10:35		ID:Y0323	SDG:45454 Matrix:	W	Containers: 5
MS320	4	01/09/2001	ABC	EPA - 25 ML			
MS519	4	01/09/2001	DE	BNA BY OLM04.2			
Sample: 45454.07		Sampled:12/19/00 11:50		ID:Y0324	SDG:45454 Matrix:	W	Containers: 5
MS320	4	01/09/2001	ABC	EPA - 25 ML			
MS519	4	01/09/2001	DE	BNA BY OLM04.2			
Sample: 45454.08		Sampled:12/19/00 12:05		ID:Y0325	SDG:45454 Matrix:	W	Containers: 5
MS320	4	01/09/2001	ABC	EPA - 25 ML			
MS519	4	01/09/2001	DE	BNA BY OLM04.2			
Sample: 45454.09		Sampled:12/19/00 13:15		ID:Y0326	SDG:45454 Matrix:	W	Containers: 5
MS320	4	01/09/2001	ABC	EPA - 25 ML			
MS519	4	01/09/2001	DE	BNA BY OLM04.2			
Sample: 45454.10		Sampled:12/19/00 13:50		ID:Y0327	SDG:45454 Matrix:	W	Containers: 5
MS320	4	01/09/2001	ABC	EPA - 25 ML			
MS519	4	01/09/2001	DE	BNA BY OLM04.2			
Sample: 45454.11		Sampled:12/19/00 15:00		ID:Y0328	SDG:45454 Matrix:	W	Containers: 5
MS320	4	01/09/2001	ABC	EPA - 25 ML			
MS519	4	01/09/2001	DE	BNA BY OLM04.2			

ORIGINAL DOCUMENTS ARE INCLUDED IN
CSF 28757 SDG Y0228
Signature S. Jackett
Date 12/22/00

ATTACHMENT 5
Client / Laboratory Communication Form

CLIENT/LABORATORY COMMUNICATION FORM, SWL-GA-131

CLIENT LABORATORY COMMUNICATION FORM (131-fig4.doc), SWL SOP REV. 1.0, FORM ID: GA-131-COMM-F

➤ In Reference to Case Contract/Proposal:

TELEPHONE RECORD LOG

➤ Date of Call:

➤ Client Name:

➤ Client Contact:

➤ Call Initiated By: ☐ Client ☐ Laboratory

➤ In reference to data for the following sample number(s):

➤ Sumary of Questions/Issues Discussed:

➤ Summary of Resolution:

Signature: *Date:*

➤ Distribution: ✔ White/Lab Copy ✔ Yellow/Client Copy ✔ Pink/Project Officer Copy

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

ATTACHMENT 6
Facility Transfer & Analytical Services Request Form

Facility Transfer & Analytical Services Request Form

SWL-GA-134; Form Name: GA134-Trans-F; Path and Filename: F:\Navy97\Series\G-Series\Attchmnt\Ga-134-1

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. / American Analytical & Technical Services, Inc.
1700 W. Albany • Broken Arrow, Oklahoma 74012 • Office (918) 251-2858 • Fax (918) 251-2599

ATTACHMENT 7
Outside Analytical Services Request

OUTSIDE ANALYTICAL SERVICES REQUEST

SAMPLE RECEIVING DEPARTMENT

OUTSIDE ANALYTICAL SERVICES REQUEST (110-ATT8.WB1) SWL-GA-110 Rev. 3.0 8/22/97 BOOK # GA-110-OUTANSRVREQ

™

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]

Typist: LLR

ATTACHMENT 8
Internal COC

SOUTHWEST LABORATORY OF OKLAHOMA, INC.

INTERNAL COC

Client: EPAMT136 Project: 25653
Episode: 30817

Test Code	Cont ID	Samp. #	Samp. #	Samp. #	Samp. #	Samp. #	Samp. #	Location (ref/shelf)	Date/Time Out	Analyst	Date/time In
IN120	A	7	9								
	B	1	2	6	8						
	C	5						H9	9/8 11:00		9/8 100
	D	5									
MT310	A	1 8	2	3	4	5	6				
	B	5									
MT600	A	1 8	2	3	4	5	6				
	B	5							9-8/600	JDL	9-8/330
MT620	A	1 8	2	3	4	5	6				
	B	5							9-9 6:00		9-9/330
MT810	A	1 8	2	3	4	5	6				
	B	5									
MT813	A	1 8	2	3	4	5	6				
	B	5									

ATTACHMENT 9
Sample Modification or Cancellation Request

SAMPLE MODIFICATION OR CANCELLATION REQUEST, SWL-GA-171

Sample Modification or Cancellation Request (171-fig5.doc), SWL SOP Rev. 1.0, Form ID: GA-171-Mod-F

To be completed by the Requesting Individual

NAME:	DATE:
DEPARTMENT:	

REQUEST TYPE:	Modification ☐	Cancellation ☐
EXPLANATION OF REQUEST:		

AUTHORIZATION BY PROJECT OFFICER:	DATE:

To be completed by Sample Receiving

PROJECT COMPLETED:

BY:	DATE:

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

ATTACHMENT 10
Refrigerator Temperature Record

SWLO / AATS

REFRIG: _____
MONTH: _____
THERM. ID: _____

REFRIGERATOR TEMPERATURE RECORD, SWL-GA-128-1
File Name (F:\Navy97\Sop's\Forms\Books\GA128F1.doc) SWL SOP Rev.2.1 Book ID: GA-128-ZoneRef-3

CONTROL LIMITS: 2.0 °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

TYPIST: LLR

ATTACHMENT 11
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 12
Archive Record Sheet

SOUTHWEST LABORATORY OF OKLAHOMA, INC.

ARCHIVE RECORD SHEET (110-ATT7.WB1) SWL-GA-110 Rev. 3.0 BOOK # GA-110-ARCHRECRD

Page 1

SAMPLES READY TO BE ARCHIVED

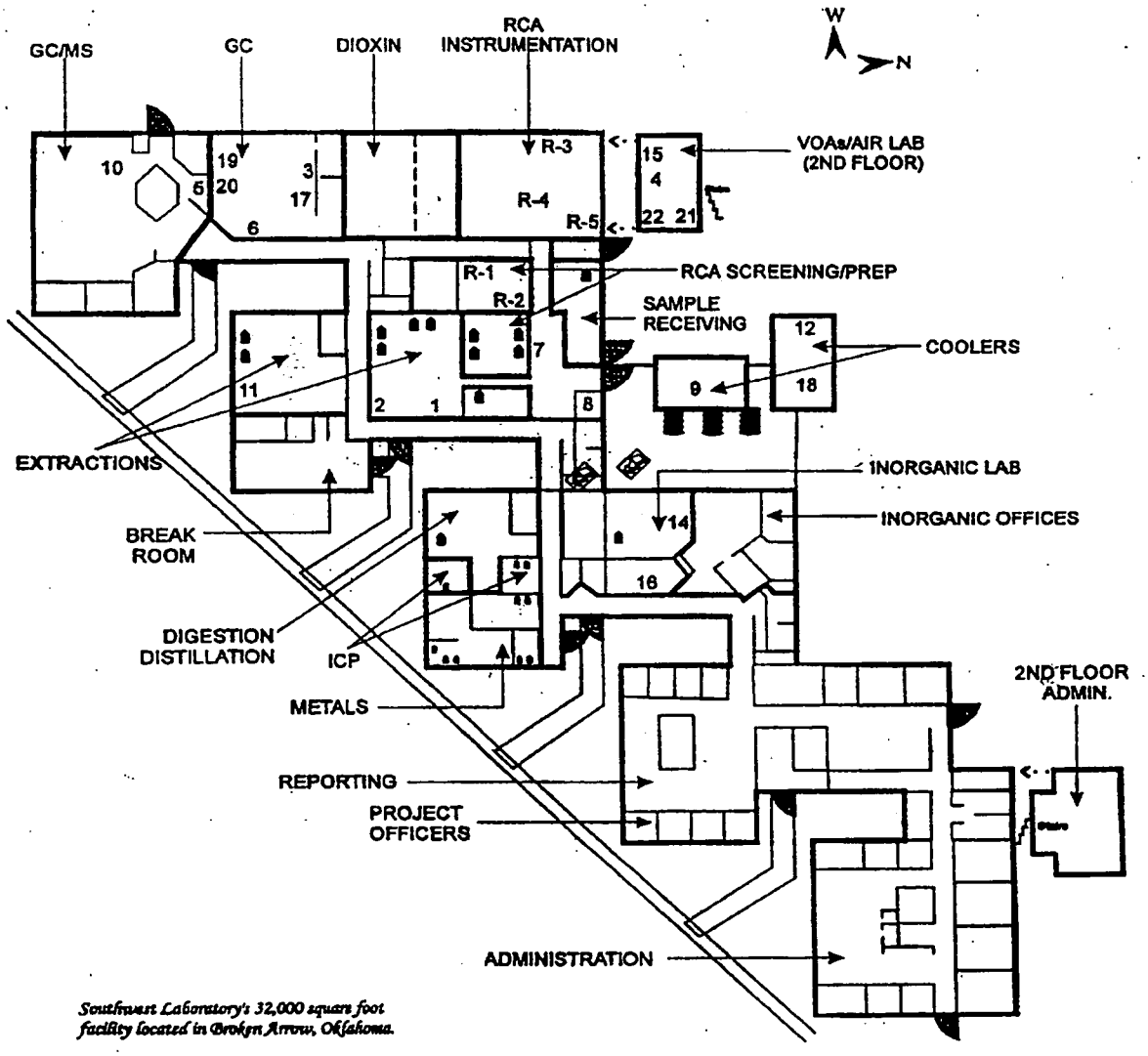
ARCHIVE DATE 09-13-97
DISPOSAL DATE 12-12-97

SAMPLE	CLIENT	DESCRIPTION	MATRIX	LOGIN
30061.01	EPA	62-SO-MNW1-01	S	07/12/97
30061.02	EPA	62-SO-MNW1-101	S	07/12/97
30061.03	EPA	62-SO-MNW1-02	S	07/12/97
30061.04	EPA	EB0711971	W	07/12/97
30061.05	EPA	TB0711971	W	07/12/97
30077.01	EPA14	62-SO-MNW2-01	S	07/14/97
30077.02	EPA14	62-SO-MNW2-02	S	07/14/97
30077.03	EPA14	TB0713971	W	07/14/97
30077.04	EPA14	EB0713971	W	07/14/97
30077.05	EPA14	62-SO-MNW3-01	S	07/14/97
30077.06	EPA14	62-SO-MNW3-02	S	07/14/97
30099.06	EPA14	TB0714971	W	07/15/97
30099.07	EPA14	AB0714971	W	07/15/97
30099.08	EPA14	62-SO-MNW4-01	S	07/15/97
30367.01	AATS-E	EAFN1 (CASE#25602)	S	07/30/97
30367.02	AATS-E	EAFN2 *QA/QC	S	07/30/97
30367.03	AATS-E	EAFN3 (CASE#25602)	S	07/30/97
30367.04	AATS-E	EAFN4 (CASE#25602)	S	07/30/97
30367.05	AATS-E	EAFN5 (CASE#25602)	S	07/30/97
30367.06	AATS-E	EAFN6 (CASE#25602)	S	07/30/97
30367.07	AATS-E	EAFN7 (CASE#25602)	S	07/30/97
30367.08	AATS-E	EBXZ0 (CASE#25602)	S	07/30/97
30367.09	AATS-E	EBXZ1 (CASE#25602)	S	07/30/97
30367.10	AATS-E	EBXZ2 (CASE#25602)	S	07/30/97
30367.11	AATS-E	EBXZ3 (CASE#25602)	S	07/30/97
30367.12	AATS-E	EBXZ4 (CASE#25602)	S	07/30/97
30367.13	AATS-E	EBXZ5 (CASE#25602)	S	07/30/97
30409.01	AATS-E	EBXZ6 (CASE#25602)	S	08/01/97
30409.02	AATS-E	EBXZ7 (CASE#25602)	S	08/01/97
30409.03	AATS-E	EBXZ8 (CASE#25602)	S	08/01/97
30409.04	AATS-E	EBXZ9 (CASE#25602)	S	08/01/97
30409.05	AATS-E	EBYA0 (CASE#25602)	S	08/01/97
30409.06	AATS-E	EBYA1 (CASE#25602)	S	08/01/97
30409.07	AATS-E	EBYA2 (CASE#25602)	S	08/01/97
30409.08	AATS-E	EBYA3 (CASE#25602)	S	08/01/97
30409.09	AATS-E	EBYA4 *QA/QC	S	08/01/97
30409.10	AATS-E	EBYA5 (CASE#25602)	S	08/01/97
30409.11	AATS-E	EBYA6 (CASE#25602)	S	08/01/97
30409.12	AATS-E	EBYA7 (CASE#25602)	S	08/01/97
30409.13	AATS-E	EBYA8 (CASE#25602)	S	08/01/97
30409.14	AATS-E	EBYA9 (CASE#25602)	S	08/01/97
30409.15	AATS-E	EBYB0 (CASE#25602)	S	08/01/97
30409.16	AATS-E	EBYB1 (CASE#25602)	S	08/01/97
30409.17	AATS-E	EBYB2 (CASE#25602)	S	08/01/97
30409.18	AATS-E	EBYB3 (CASE#25602)	S	08/01/97
30409.19	AATS-E	EBYB4 (CASE#25602)	S	08/01/97
30409.20	AATS-E	EBYB5 (CASE#25602)	S	08/01/97
30409.21	AATS-E	EBYB6 (CASE#25602)	S	08/01/97
30409.22	AATS-E	EBYB7 (CASE#25602)	S	08/01/97

ATTACHMENT 13
Facility Layout

Southwest Laboratory of Oklahoma, Inc. • 1700 W. Albany • Broken Arrow, OK • 251-2858

FACILITY LAYOUT



Southwest Laboratory's 32,000 square foot facility located in Broken Arrow, Oklahoma.

Note: The numbers indicate the refrigerator or freezer number.

Facility Layout: GA123 021902 - GA115 042302

ATTACHMENT 14
FedEx® Package Tracking Summary

FedEx. 12OCT 08:15 Pcs: 19 Emp#: 419183 1 of 2 Rt#: 653
Recv'd by: J. SPRIGGS
Pkg Trk#'s: 455770054194

792455845760	791677091646
3206817117	830045419249
3206812567	826867902505
421553557054	790183876779
830696716500	829729112201
828779514729	3220775966
421553557065	811011702178
421553557043	455770054210

FedEx. 12OCT 08:15 Pcs: 19 Emp#: 419183 2 of 2 Rt#: 653
Recv'd by: J. SPRIGGS

455770054209	3206843954

10-12-01

Pace Analytical®
www.pacelabs.com

Pace Analytical Services, Inc
1700 West Alban
Broken Arrow, OK 7401.
Phone: 918.251.285.
Fax: 918.251.259.

GA-116 / -120 / -121

The Standard Operating Procedures (SOPs) contained herein are adopted by Pace Analytical Services.

These SOPs will be followed until superceded by Pace Analytical Services SOPs.

Signature:

Chuck Hoover
Quality Assurance Officer
Pace Analytical Services, Inc.
Broken Arrow, Oklahoma

Signature:

Roger C. Splinter
Dir. of Quality, Safety, Training
Pace Analytical Services, Inc.
Minneaplolis, MN 55414

Signature:

Dave Neal
General Manager
Pace Analytical Services, Inc.
Lenexa, KS

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

Standard Operating Procedure

Bottle Preparations

Date: Rev. 4.0 08/01/02

Rev.

OFFICIAL COPY
RED STAMP INDICATES ORIGINAL

DATES

8-5-02

APPROVALS

BOTTLE PREPARATIONS

TABLE OF CONTENTS

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5.4 Bottle Order Completion 2
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Attachment 1B: Recommendation for Sampling and Preservation of Samples According to Measurement 5
Attachment 2: Copies of Airbills (Figure 1 – Figure 3) ..... 6 - 7

1.0 PURPOSE AND APPLICATION

1.1 This procedure is used for meeting the client's need for bottles. The client utilizes these bottles during sampling of various matrices in the field.

1.2 Over the course of a particular project, clients will call the project officer with a bottle request. A bottle request form is filled and sent to the shipping and receiving department where the bottle order is filled and sent out to the client.

2.0 RESPONSIBILITIES

2.1 It is the responsibility of the Project Officer and/or the Marketing Representative to initiate the bottle request.

2.2 It is the responsibility of the Shipping & Receiving Department Personnel to fill the bottle order and properly ship items requested to the client.

3.0 EQUIPMENT

Not applicable.

4.0 PRECAUTIONS

See MSDS for hazards related to the preservatives used.

5.0 PROCEDURE

5.1 SWLO uses bottles supplied by I-Chem and/or ESS. Certified bottles are available should the client require the documentation. The bottle shipment personnel maintain all documentation of certification.

5.2 Preservatives are prepared in the Inorganic or Volatile Organic departments. See SOP SWL-IN-104 for details on preparations. When these preservatives are prepared, lot numbers are given to them; they are checked for purity prior to use. Table 1 lists the preservatives used in bottle preparation.

Table 1

Preservative	Concentration	Parameters	Amt. Of Preservatives/ Container
Hydrochloric Acid (HCL)	1:1	Volatile Organics	0.5ml/40ml vial
Nitric Acid (HNO_3)	1:1	Metals	2.0ml/L
Nitric Acid (HNO_3)	1N	Radiologicals (Except Tritium)	2.0ml/L
Sulfuric Acid (H_2SO_4)	1:1	Nitrate/Nitrite, Phenols, TOX, TOC, COD	2.0ml/L
Sodium Hydroxide (NaOH)	10N	Sulfide Cyanide	2.0ml/L
Zinc Acetate Sodium Thiosulfate ($Na_2S_2O_3$)	2N 0.008%	Sulfide Chlorinated Drinking Water Organics	1.0ml/L 1.0ml/L

5.3 Bottle Order Initiation — During a project, the client will periodically call a project officer or marketing representative with a request for bottles. The order is taken and a Client Shipping Containers Request (commonly referred to as Container Request Form — see Attachment 1) is filled out (either standard or priority, using a hardcopy or online form). Information is recorded including client name, client contact, project name, requested carrier, shipping address, required delivery date, quantity, matrix, bottle type, preservative, etc.

5.3.1 Hardcopy Form — Most container requests will be completed using the "online" version of this form; however, the form may occasionally be completed by hand. All applicable information must be included in the form.

Note: The laboratory personnel completing the form (project officers, marketing, and management) must choose the appropriate container (polyethylene, glass, amber glass, or glass with or without preservative) for the specific analysis requested. See Attachment 1B for sample preservation and bottle information. In addition, all applicable sections of the form must be completed as specifically as possible.

5.3.2 Online Form — Most container requests will be completed using the "online" version of this form (see section 5.3.1 for hardcopy use).

Note: The laboratory personnel completing the form (project officers, marketing, and management) must choose the appropriate container (polyethylene, glass, amber glass, or glass with or without preservative) for the specific analysis requested. See Attachment 1B for sample preservation and bottle information. In addition, all applicable sections of the form must be completed as specifically as possible.

The online form is a document template titled "Ga116A1.dot" located in "S:\Sample Receiving and Shipping" and can be renamed using the following naming convention and saved in the subfolder "Completed Form Ga116A1."

Client Code + Date = EKI-CA 082102

and for multiple requests on the same day:
Client Code + Date + -1, -2, etc. = EKI-CA 082102-1

5.4 Bottle Order Completion — The project officer delivers the Container Request Form to the Shipping & Receiving department where the bottles are prepared.

5.4.1 Bottles are prepared in accordance with the Container Request Form.

5.4.1.1 If preservatives are required, lot numbers are recorded on the Container Request Form.

5.4.1.2 All applicable Material Safety Data Sheets (MSDS) are included in the sample shuttle for preservatives used.

5.4.1.3 Bottle lids are taped (to prevent leakage).

5.4.1.4 Bottles are marked with appropriate labels indicating preservative used.

5.4.1.5 Bottles are packaged (normally in coolers) and shipped out.

5.4.2 The type of shipment, Tracking Number, preservatives/container lot numbers used, and all other pertinent information is recorded on the Container Request Form for future tracking purposes.

5.4.3 The Container Request Form is reviewed and documented (with initials and date) by a verifier.

6.0 RECORDS

6.1 Container Request Forms (Attachment 1) are filed in sample receiving for reference.

6.2 Copies of Airbills are maintained with original paperwork to track shipments. (See Attachment 2).

7.0 REFERENCES

Not applicable.

8.0 DEFINITIONS

Not applicable.

9.0 ATTACHMENTS

Attachment 1: Client Shipping Containers Request
Attachment 1B: Recommendation for Sampling and Preservation of Samples According to Measurement
Attachment 2: Copies of Airbills

ATTACHMENT 1
Client Shipping Containers Request

CLIENT SHIPPING CONTAINERS REQUEST

DEPARTMEN

Bottle Preparations SWL-GA-116, 4.0 08/02/02 File ID: GA116A1.dot White Copy — Accounting • Yellow Copy — Client • Pink Copy — File

☐ RUSH

Client Code	Project Name	Date Contacted	Required Ship Date	For Delivery By	Shipper Requested

CLIENT INFORMATION

Client: _____

Client Contact: _____

Phone No.: _____

Shipping Address: _____

Containers: ☐ Billable ☐ Not Billable

If NOT billable, please explain:

(RUSH Shipment Only)

Shipping: ☐ Billable ☐ Not Billable

If NOT billable, please explain:

SWL Contact:

Approved By:

SHIPPING INFORMATION

☐ Coolers (QTY. _____) ☐ Blue Ice ☐ Return Shipping Labels ☐ Make Bottle Labels

☐ Custody Seals (QTY. _____) ☐ COC's ☐ Enclose Blank Bottle Labels ☐ Attach Bottle Labels

MISC./SPECIAL PROVISIONS:

CONTAINER REQUIREMENTS

✓	QTY.	MATRIX	CONTAINERS	CONTAINER LOT #	ANALYTICAL	PRESERVATIVE	PRESERVATIVE LOT
☐							
☐							
☐							
☐							
☐							
☐							
☐							
☐							
☐							
☐							
☐							
☐							
☐							
☐							
☐							

DATE SHIPPED	# PIECES	TOTAL WEIGHT	FILLED BY	SHIPPED VIA	SHIPPERS REF. #

Southwest Laboratory of Oklahoma, Inc.
1700 West Albany • Broken Arrow, OK 74012 • Office (918) 251-2858 • Fax (918) 251-2599

ATTACHMENT 1B
Recommendation for Sampling and Preservation of Samples According to Measurement

Recommendation For Sampling And Preservation Of Samples According To Measurement[1]

Measurement	Vol. Req. (mL)	Holding Container[2]	Preservative[3,4]	Time[5]
Physical Properties				
Color	50	P, G	Cool, 4°C	48 Hrs.
Conductance	100	P, G	Cool, 4°C	28 days
Hardness	100	P	HNO_3 to pH <2	6 mos.
Odor	200	G only	Cool, 4°C	24 Hrs.
pH	2	P, G	None Required,	Analyze Immediately
Residue				
Filterable	100	P, G	Cool, 4°C	7 days
Non-Filterable	100	P, G	Cool, 4°C	7 days
Total	100	P, G	Cool, 4°C	7 days
Volatile	100	P, G	Cool, 4°C	7 days
Settleable Matter	1,000	P, G	Cool, 4°C	48 Hrs.
Temperature	1,000	P, G	None Req.	Analyze Immediately
Turbidity	100	P, G	Cool, 4°C	48 Hrs.
Metals				
Dissolved	200	P	Filter on site HNO_3 to pH <2	6 Mos.
Suspended	200		Filter on site	6 Mos.
Total	100	P	HNO_3 to pH <2	6 Mos.
Chromium^{+6}	200	P	Cool, 4°C	24 Hrs.
Mercury (Dissolved)	100	P	Filter HNO_3 to pH <2	28 Days
Mercury (Total)	100	P	HNO_3 to pH <2	28 Days
Inorganics, Non-Metallics				
Acidity	100	P, G	Cool, 4°C	14 Days
Alkalinity	100	P, G	Cool, 4°C	14 Days
Bromide	100	P, G	None Req.	28 Days
Chloride	50	P, G	None Req.	28 Days
Chlorine	200	P, G	None Req.	Analyze Immediately
Cyanides	500	P, G	Cool, 4°C NaOH to pH >12 0.6g ascorbic acid[6]	14 Days[7]
Fluoride	300	P, G	None Req.	28 Days

Measurement	Vol. Req. (mL)	Holding Container[2]	Preservative[3,4]	Time[5]
Inorganics, Non-Metallics				
Iodide	100	P, G	Cool, 4°C	24 Hrs.
Sulfate	50	P, G	Cool, 4°C	28 Days
Silica	50	P only	Cool, 4°C	28 Days
Sulfide	500	P, G	Cool, 4°C add 2 mL zinc acetate plus NaOH to pH >9	7 Days
Sulfite	50	P, G	None Req.	Analyze Immediately
Nitrogen				
Ammonia	400	P, G	Cool, 4°C H_2SO_4 to pH <2	28 Days
Kjeldahl, Total	500	P, G	Cool, 4°C H_2SO_4 to pH <2	28 Days
Nitrate plus Nitrite	100	P, G	Cool, 4°C H_2SO_4 to pH <2	28 Days
Nitrate[9]	100	P, G	Cool, 4°C	48 Hrs.
Nitrite	50	P, G	Cool, 4°C	48 Hrs.
Dissolved Oxygen				
Probe	300	G bottle and top	None Req.	Analyze Immediately
Winkler	300	G bottle and top	Fix on site and store in dark	8 Hrs.
Phosphorus				
Orthophosphate, Dissolved	50	P,G	Filter on site Cool, 4°C	48 Hrs.
Hydrolyzable	50	P,G	Cool, 4°C H_2SO_4 to pH <2	28 Days
Total	50	P,G	Cool, 4°C H_2SO_4 to pH <2	28 Days
Total, Dissolved	50	P,G	Filter on site	24 Hrs.
Organics				
BOD	1,000	P,G	Cool, 4°C	48 Hrs.
COD	50	P,G	Cool, 4°C H_2SO_4 to pH <2	28 Days
Oil & Grease	1,000	G only	Cool, 4°C H_2SO_4 or HCl to pH <2	28 Days
Organic Carbon	25	P,G	Cool, 4°C H_2SO_4 or HCl to pH <2	28 Days

Measurement	Vol. Req. (mL)	Holding Container[2]	Preservative[3,4]	Time[5]
Organics				
Phenolics	500	G only	Cool, 4°C H_2SO_4 or HCl to pH <2	28 Days
MBAS	250	P,G	Cool, 4°C, 48 Hrs.	
NTA	50	P,G	Cool, 4°C 24 Hrs.	
Purgeable Halocarbons (601)	40	G, Teflon-lined Septum	Cool, 4°C, 14 Days 0.008% $Na_2S_2O_3$[6]	
Purgeable Aromatic Hydrocarbons (602)	40	G, Teflon-lined Septum	Cool, 4°C, 0.008% $Na_2S_2O_3$[6], HCl to pH <2	10 Days[10]
Acrylonitrile & Acrolein (603)	40	G, Teflon-lined Septum	Cool, 4°C, 0.008% $Na_2S_2O_3$[6], Adjust pH to 4-5[11]	14 Days
Phenols[12] EPA Method 604	1,000	G, Teflon-lined Cap	Cool, 4°C, 0.008% $Na_2S_2O_3$	7 Days until extraction, 40 days after extraction
Benzidines[12] EPA Method 605	1,000	G, Teflon-lined Cap	Cool, 4°C, 0.008% $Na_2S_2O_3$[6], H_2SO_4 to pH 2-7[13]	7 days until extraction
Phthalate Esters[12] EPA Method 606	1,000	G, Teflon-lined Cap	Cool, 4°C	7 days until extraction, 40 days after extraction
Nitrosamines[12] EPA Method 607	1,000	G, Teflon-lined Cap	Cool, 4°C, store in dark, 0.008% $Na_2S_2O_3$[6]	7 days until extraction, 40 days after extraction
Organochlorine Pesticides/PCBs[12] EPA Method 608 8081A/8082	1,000	G, Teflon-lined Cap	Cool, 4°C, pH 5-9	7 days until extraction, 40 days after extraction
Nitroaromatics and Isophorone[12] EPA Method 609	1,000	G, Teflon-lined Cap	Cool, 4°C, 0.008% $Na_2S_2O_3$[6], store in dark	7 days until extraction, 40 days after extraction
Polynuclear Aromatic Hydrocarbons[12] EPA Method 610/8310	1,000	G, Teflon-lined Cap	Cool, 4°C, 0.008% $Na_2S_2O_3$[6] store in dark	7 days until extraction, 40 days after extraction

Measurement	Vol .Req. (mL)	Holding Container[2]	Preservative[3,4]	Time[5]
Organics (continued)				
Haloethers[12] EPA Method 611	1,000	G, Teflon-lined Cap	Cool, 4°C, 0.008% $Na_2S_2O_3$	7 days until extraction, 40 days after extraction
Chlorinated Hydrocarbon[12] (612)	1,000	G, Teflon-lined Cap	Cool, 4°C	7 days until extraction, 40 days after extraction
TCDD[12] EPA Method 1613 8280A/8290	1,000	G, Teflon-lined Cap	Cool, 4°C, 0.008% $Na_2S_2O_3$[6]	7 days until extraction, 40 days after extraction extraction
Purgeables EPA Method 624/601/602 8021B/8260B	2 x 40	G, Teflon-lined Septum	Cool, 4°C, 0.008% $Na_2S_2O_3$[6]	14 Days
Base/Neutrals Acids EPA Method 625/8270C	1,000	G, Teflon-lined Cap	Cool, 4°C, 0.008% $Na_2S_2O_3$[6]	7 days until extraction, 40 days after extraction
Radiochemistry				
Tritium	200	Glass	None	6 months
All others	1000	P,G	HNO_3 to pH <2	6 months

Footnotes:

1 More specific instructions for preservation and sampling are found with procedures as detailed in EPA-600/4-79-020, revised March, 1983, and in the Federal Register, Vol. 49, No. 209, Oct. 26, 1984, EPA 40 CFR part 136.

2 Plastic (P) or glass (G). Only EPA certified pre-cleaned bottles are used. For metals, polyethylene with a polypropylene cap (no liner) is preferred.

3 Sample preservation should be performed immediately upon sample collection. For composite samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then samples may be preserved by maintaining them at 4°C until compositing and sample splitting is completed.

4 When any sample is to be shipped by common carrier or sent through the US Mail, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements of Table 1, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: (1) Hydrochloric Acid (HCl) in water solutions at concentrations of % by weight or less (pH about 1.96 or greater). (2) Nitric Acid (HNO_3) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater). (3) Sodium Hydroxide (NaOH) in water solutions at concentrations of 0.08 by weight or less (pH about 12.3 or less).

5 Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still considered valid. Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that the specific types of sample under study are stable for the longer time and has received a variance from the Regional Administrator. Some samples may not be stable for the maximum time period given in the table. A permittee, or monitoring

laboratory, is obligated to hold the sample for a shorter time if knowledge exists to show that this is necessary to maintain sample stability.

6 Should only be used in the presence of residual chlorine.

7 Maximum holding time is 24 hours when sulfide is present; optionally, all samples may be tested with lead acetate paper before the pH adjustment in order to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.

8 Samples should be filtered immediately on-site before adding preservatives for dissolved metals.

9 For samples from non-chlorinated drinking water supplies, concentrated H_2SO_4 should be added to lower sample pH to less than 2. The sample should be analyzed before 14 days. Samples receiving no pH adjustment must be analyzed within seven days of sampling.

10 The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of the sampling.

11 When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4°C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6 – 9. Samples preserved in this manner may be held for 7 days before extraction and 40 days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 6 (requirement for thiosulfate reduction of residual chlorine).

12 If 1,2-Diphenylhydrazine is likely to be present, adjust the sample pH to 4.0 ± 0.2 to prevent rearrangement to benzidine.

Measurement	Container Size (oz)	Container	Holding Preservative	Time
Inorganics, Non-Metallics				
Petroleum Hydrocarbons	32	G	Cool, 4°C	28 da
Metals				
Total Recoverable	8	G	Cool, 4°C	6 months
Mercury (total)	8	G	Cool, 4°C	28 days
Extraction Procedure Toxicity	32	G	Cool, 4°C	6 months
Organics				
VOCs and Xylenes, EPA Method 8260B	2 x 4	G	Cool, 4°C	14 days
Extractable Priority Pollutants EPA Method 8270C	32	G	Cool, 4°C	14 days – extraction, 40 days – analysis

A More specific instructions for preservation and sampling are found with procedures as detailed in SW846, "Test methods for Evaluating Solid Waste, Physical/Chemical Methods", 3rd Edition, Chapter 4.

ATTACHMENT 2
Copies of Airbills

FedEx. Federal Express

USE THIS AIRBILL FOR SHIPMENTS WITHIN THE CONTINENTAL U.S.A., ALASKA AND HAWAII.
USE THE INTERNATIONAL AIR WAYBILL FOR SHIPMENTS TO PUERTO RICO AND ALL NON U.S. LOCATIONS.
QUESTIONS? CALL 800-238-5355 TOLL FREE.

AIRBILL
PACKAGE TRACKING NUMBER 5553037630

5163M 5553037630

43f
9485

SENDER'S FEDERAL EXPRESS ACCOUNT NUMBER: 1222-5116-0
Date

From (Your Name) Please Print
Your Phone Number (Very important): (918)251-2858
Company: SOUTHWEST LABORATORIES
Department/Floor No.
Street Address: 1700 W ALBANY STE C
City: BROKEN ARROW State: OK ZIP Required: 74012

To (Recipient's Name) Please Print
Recipient's Phone Number (Very important): ( )
Company
Department/Floor No.
Exact Street Address (We Cannot Deliver to P.O. Boxes or P.O. Zip Codes.)
City State ZIP Required

215
1000

YOUR INTERNAL BILLING REFERENCE INFORMATION (optional) (First 24 characters will appear on invoice.)

IF HOLD AT FEDEX LOCATION, Print FEDEX Address Here
Street Address
City State ZIP Required

PAYMENT 1 ☐ Bill Sender Acct. No. Req'd. 2 ☐ Bill Recipient's FedEx Acct. No. Fill in Account Number below 3 ☐ Bill 3rd Party FedEx Acct. No. Fill in Account Number below (req'd.) 4 ☐ Bill Credit Card Fill in Credit Card No. below (req'd.) 5 ☐ Cash/Check
Acct./Credit Card No. Exp. Date

SERVICES (Check only one box)
Priority Overnight (Delivery by next business morning!): 11 ☐ OTHER PACKAGING; 16 ☐ FEDEX LETTER*; 12 ☐ FEDEX PAK*; 13 ☐ FEDEX BOX; 14 ☐ FEDEX TUBE
Standard Overnight (Delivery by next business afternoon, No Saturday delivery!): 51 ☐ OTHER PACKAGING; 56 ☐ FEDEX LETTER*; 52 ☐ FEDEX PAK*; 53 ☐ FEDEX BOX; 54 ☐ FEDEX TUBE
Economy Two-Day (Delivery by second business day!): 30 ☐ ECONOMY**
Government Overnight (Restricted for authorized users only): 46 ☐ GOVT LETTER; 41 ☐ GOVT PACKAGE
*Economy Letter Rate not available. Minimum charge: One pound Economy rate.
Freight Service (for packages over 150 lbs.): 70 ☐ OVERNIGHT FREIGHT** (Confirmed reservation required); 80 ☐ TWO-DAY FREIGHT**
†Delivery commitment may be later in some areas.
*Declared Value Limit $500. **Call for delivery schedule.

DELIVERY AND SPECIAL HANDLING (Check services required)
Weekday Service: 1 ☐ HOLD AT FEDEX LOCATION WEEKDAY (Fill in Section H); 2 ☐ DELIVER WEEKDAY
Saturday Service: 31 ☐ HOLD AT FEDEX LOCATION SATURDAY (Fill in Section H); 3 ☐ DELIVER SATURDAY (Extra charge) (Not available to all locations); 9 ☐ SATURDAY PICK-UP (Extra charge)
Special Handling: 4 ☐ DANGEROUS GOODS (Extra charge); 6 ☐ DRY ICE (Dangerous Goods Shipper's Declaration not required); 12 ☐ HOLIDAY DELIVERY (If offered) (Extra charge)

PACKAGES | WEIGHT In Pounds Only | YOUR DECLARED VALUE (See right)
Total | Total | Total

DIM SHIPMENT (Chargeable Weight) ☐ lbs.
L x W x H

Received At: 1 ☐ Regular Stop 2 ☐ On-Call Stop 3 ☐ Drop Box 4 ☐ B.S.C. 5 ☐ Station

SERVICE CONDITIONS, DECLARED VALUE AND LIMIT OF LIABILITY
Use of this airbill constitutes your agreement to the service conditions in our current Service Guide, available upon request. See back of sender's copy of this airbill for information. Service conditions may vary for Government Overnight Service. See U.S. Government Service Guide for details.
We will not be responsible for any claim in excess of $100 per package, whether the result of loss, damage, delay, non-delivery, misdelivery, or misinformation, unless you declare a higher value, pay an additional charge, and document your actual loss for a timely claim. Limitations found in the current Federal Express Service Guide apply. Your right to recover from Federal Express for any loss, including intrinsic value of the package, loss of sales, income interest, profit, attorney's fees, costs, and other forms of damage whether direct, incidental, consequential, or special is limited to the greater of $100 or the declared value specified to the left. Recovery cannot exceed actual documented loss. The maximum Declared Value for FedEx Letter and FedEx Pak packages is $500.
In the event of untimely delivery, Federal Express will at your request and with some limitations refund all transportation charges paid. See Service Guide for further information.
Sender authorizes Federal Express to deliver this shipment without obtaining a delivery signature and shall indemnify and hold harmless Federal Express from any claims resulting therefrom.
Release Signature:

Federal Express Use
Base Charges
Declared Value Charge
Other 1
Other 2
Total Charges

REVISION DATE 4/94 PART #145413 QBFE 5/95 FORMAT #160
160
© 1993-94 FEDEX PRINTED IN U.S.A.

FEDEX COPY

Figure 1: FedEx Airbill

ATTACHMENT 2
Copies of Airbills

US Delivery

U. S. DELIVERY SYSTEMS - SOUTHWEST, INC.

400 N. MACARTHUR OKC, OK 73127 405-946-0099 | 348 N. GUAPAW OKC, OK 73189 405-946-2085 | HWY. 69 N. McALESTER, OK 74501 918-423-1194 | 428 S. 122nd EAST AVENUE TULSA, OK 74116 918-437-7117 | 1607 W. CHESTNUT ENID, OK 73703 405-234-4001

DELIVERY RECEIPT

ACCOUNT NO.

AIRBILL NUMBER 300-6543519

ROUTE NUMBERS

CUSTOMER REF. NO.

TOTAL CHARGES $

COD PRODUCT

COD FEE

DECLARED VALUE

INSUR FEE

DENT FEE

SHIPPER DECLARES REQUIREMENT FOR RAPID MOVEMENT NOT AVAILABLE FROM CLASS A CARRIERS

FROM: COMPANY NAME LOC. NO.
ADDRESS:
CITY: STATE: ZIP:
CONTACT: PHONE:

TO: COMPANY NAME LOC. NO.
ADDRESS: (NO P.O. BOX ALLOWED)
CITY: STATE: ZIP (REQ)
CONTACT: PHONE:

THIS PACKAGE IS ACCEPTED BY US DELIVERY FOR SHIPMENT BASED ON THE TERMS AND CONDITIONS ON THE REVERSE SIDE OF THIS AIRBILL.

DESCRIBE CONTENTS:

ADDITIONAL DELIVERY INSTRUCTIONS:

SHIPPER SIGNATURE DATE: X TIME:

FOR U.S. DELIVERY DATE: X TIME:

PIECES | WEIGHT

PAYMENT TERMS ☐ PREPAID ☐ COLLECT ☐ CASH

RECEIVED IN GOOD ORDER AND CONDITION DATE:
PLEASE PRINT NAME X TIME:

Figure 2: U.S. Delivery Systems

AIRBORNE EXPRESS

PLEASE TYPE OR PRINT — U.S. AIRBILL

1 FROM (COMPANY NAME): SOUTHWEST LABS
ADDRESS: STE A 1700 W ALBANY
CITY: BROKEN ARROW STATE: OK ZIP CODE (REQUIRED): 74012
SENT BY (NAME/DEPT.) PHONE

2 TO (COMPANY NAME)
ADDRESS
CITY STATE ZIP CODE (REQUIRED)
ATTN: (NAME/DEPT.) PHONE

ORIGIN: TUL NUMBER: 713

4 CUSTOMER AIRBORNE EXPRESS ACCOUNT NUMBERS
SENDER: 42212200
RECEIVER

5 METHOD OF PAYMENT (ASSUMED SENDER UNLESS OTHERWISE NOTED)
☐ BILL SENDER ☐ BILL RECEIVER
Airborne Account No.
☐ BILL 3RD PARTY
Check No. Amount
☐ PAID IN ADVANCE

BILLING REFERENCE (WILL APPEAR ON INVOICE)

6 NO. OF PKGS WEIGHT (LBS.) SUBJECT TO CORRECTION

7 CHECK IF ☐ LETTER EXPRESS ☐ EXPRESS PACK

SPECIAL INSTRUCTIONS
☐ SATURDAY DELIVERY ☐ HOLD AT AIRBORNE EXPRESS ☐ LAB PACK ☐ SELECT DELIVERY SERVICE

VALUE ☐ DECLARED VALUE OR ☐ FULL INSURANCE SHIPMENT VALUATION $

PREPRINT FORMAT NO. 1857014

3

THANK YOU FOR SHIPPING WITH AIRBORNE EXPRESS

SENDER'S COPY

Figure 3: Airborne Express

SOUTHWEST LABORATORY OF OKLAHOMA, INC. *and*

AMERICAN ANALYTICAL & TECHNICAL SERVICES, INC.

Standard Operating Procedure

Electronic Balance Calibration Checks

Document No.: SWL-GA-120

Rev No. / Date: Rev. 4.3 — 01/10/02

OFFICIAL COPY RED STAMP INDICATES ORIGINAL

APPROVALS	DATES
Procedure Prepared By	01/10/02 Date
Laboratory Manager or Director	1/10/02 Date
Laboratory Safety Officer	1/10/02 Date
Laboratory QA/QC Officer	1/10/02 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.: 2611 Issued to: TT NUS-PA Date: 10/17/03 QA Initials: CFH

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ELECTRONIC BALANCE CALIBRATION CHECKS

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4.0 PRECAUTIONS 1
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1.0 PURPOSE AND APPLICATION

1.1 Analytical balances of various capacities and operational mode are calibrated biannually by a licensed specialist and officially recorded as verification of performance.

1.2 Stickers documenting the calibration are placed on the balance noting the calibration data as well as the date of the next calibration. **Do NOT use the balance unless it has a certification sticker!** Certificates of calibration are maintained in the QA/QC department.

1.3 Balances are checked with known calibration weights daily and/or before use. If the reading from the calibration check is not within limits, the balance will be re-calibrated by the service person.

2.0 RESPONSIBILITIES

2.1 It is the responsibility of the Program Manager, Section Supervisor and/or Group Leader to verify that the calibration is properly being checked and documented in the Zone Log Book as per SOP SWL-GA-128, Rev. 2.1.

2.2 It is the responsibility of the Analyst who checks the calibration to verify that the certification sticker on the balance is current, and that the check is performed as per this Standard Operating Procedure and the SOP "*Maintenance of Zone Log Books*", SOP SWL-GA-128, Rev. 2.1.

3.0 EQUIPMENT

3.1 Laboratory Balances (Ohaus, Sartorius, Mettler, etc.)

3.2 Class 1 weight set

4.0 PRECAUTIONS

Care must be taken when handling the Class 1 weights: tweezers should be used. **Do Not Drop The Weight.** Dropping the weight might chip or damage the weight and introduce inaccuracy to the calibration check.

5.0 PROCEDURE

5.1 Electronic top loaders, (example, Ohaus GT400)

5.1.1 Allow a warm up time of 30 to 60 minutes after plugging in the balance.

5.1.2 Center bubble level indicator of top loader using the leveling screws.

5.1.3 Using kimwipe and/or balance brush, remove any particulates from balance pan.

5.1.4 Turn power switch to "1", the top loader will run through an "auto check".

5.1.5 A small green circle "o" appears in the bottom left corner if the system is ready.

5.1.6 Press tare once. The display will read "0.00g".

5.1.7 Calibration check

5.1.7.1 Check the calibration using weights that span the anticipated weighing episode. If beakers or other equipment are to be used in the weighing process, use them in the tare process and then check the calibration.

5.1.7.2 Place calibration weight in center of pan using forceps (never touch weights with fingers).

5.1.7.3 Read and record reading from balance in the *"Daily Balance Check Log"* of the Zone Log Book (see Attachment 1 of this SOP). Record weight to the nearest 0.01gm.

5.1.7.4 Repeat steps 5.1.7.2 & 5.1.7.3 using a second, third and fourth weight.

5.1.7.5 Document all readings in the *"Daily Balance Check Log"* of the Zone Log Book (see Attachment 1 of this SOP).

NOTE: Reading is stable when "g" appears in the display.

5.1.7.6 Note control limits recorded in the balance log. Should the weights be outside the control limits, stop the calibration and notify the supervisor or QA Officer.

5.1.8 Top loader is now ready to use.

5.1.8.1 Keep pans as clean as possible.

5.1.8.2 To tare: After reading is stable, press tare and display all zeros.

5.2 Electronic Analytical Balance, Ohaus Model AS120 (reference Ohaus Operating Manual for further detail).

5.2.1 If balance has been unplugged, allow 60 minutes for warm-up.

5.2.2 Check the center bubble level using leveling screws.

5.2.3 Briefly press "ON/TARE" to tare balance; first 8.8.8.8.8.8.8., then 0.0000 will appear.

5.2.4 Calibration Check

5.2.4.1 Check the calibration using weights that span the anticipated weighing session.

5.2.4.2 Remove all objects from the pan and close the doors.

5.2.4.3 Hold down "ON/TARE" until "CAL" appears.

5.2.4.4 Release "ON/TARE \; SPAN" will appear.

- Press "OFF MODE" and the display will show "LIN".
- Press "ON/TARE" to start the linearity calibration procedure. When "ON/TARE" is release, "C 0 g" will be displayed, indicating that no weight should be in the pan.
- Press "ON/TARE". The display will show "-C-" followed by the value of the weight that must be placed on the pan.
- Place the required weight in the pan.
- Press "ON/TARE". The display will show "-C-" momentarily, then "C" followed by the next weight to be placed on the pan. Do not disturb the balance when "-C-" is displayed.
- Place the required weight on the pan, then press "ON/TARE". The display will show "-C-" while the balance re-calibrates. When the weight on the pan is displayed along with the current indicator, the balance is re-calibrated.
- Repeatedly press "OFF" mode until "END" is displayed.
- Press "ON/TARE". When released, the balance will return to normal weighing operations.

5.2.5 Taring

5.2.5.1 Place container on pan.

5.2.5.2 Press control bar once to tare, all zeros will appear. Balance is now ready for weighing.

Maintenance for the balances is documented with a sticker on the balance dating the service and the next service due date.

6.0 RECORDS

Documentation of the balance calibration checks are maintained in each areas zone book.

7.0 REFERENCES

ASTM Procedure E617.

8.0 DEFINITIONS

Class 1:.....A classification of weights that are certified/traceable to the *National Institute of Standards and Technology* (N.I.S.T) *Standards* (*NIST Handbook 44*, published 1992).

9.0 ATTACHMENTS:

Attachment 1: Daily Balance Check Log.

ATTACHMENT 1
Daily Balance Checks

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

TM

Standard Operating Procedure

Glassware & Labware Cleaning

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GLASSWARE & LABWARE CLEANING

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1.0 PURPOSE AND APPLICATION. To ensure the integrity of the samples, steps must be taken to minimize contamination from the containers they are stored in and through the glasswares or labwares used during sample analysis. If the analyte(s) to be determined is organic in nature, then a glass container should be used. If the analyte(s) is inorganic, then a plastic or polyethylene container should be used.

2.0 RESPONSIBILITIES

2.1 Glassware Prep Technician — It is the responsibility of the glassware prep technician to ensure that glassware/labware is available for sample preparation/analysis and that the glassware is cleaned as per the following procedure to ensure minimal contamination to the sample analytical process.

2.2 Section Supervisor — It is the responsibility of the section supervisor to look for occurrence of blank contamination and alert/oversee the troubleshooting to correct the problem.

3.0 EQUIPMENT. Refer to SWL-GA-100 "*Laboratory Quality Assurance Plan*", for the equipment list for the sample prep areas.

4.0 PRECAUTIONS. Refer to the SWL-GA-111 "*Laboratory Safety Plan*" for Safety Precautions.

5.0 PROCEDURE. When both organic and inorganic substances are to be analyzed, the following procedures should be taken when cleaning glassware or labware that is to be used for sample analysis.

5.1 Metals and Wetlab Labware Washing Procedures

5.1.1 Thoroughly rinse glassware with tap water immediately after use.

5.1.2 Place rinsed glassware into plastic washtub containing detergent solution, keeping *fleakers* separate in their specified tub. Soak a minimum of 30 minutes.

5.1.3 Remove glassware and brush thoroughly.

5.1.4 Rinse 3 times with tap water (Regular glass, skip to Step 5.1.6).

5.1.5 Place *fleakers only* into washtub containing 1:9 nitric acid solution. Soak for a minimum of 30 minutes.

5.1.6 Rinse 3 times with deionized water.

5.1.7 Place upside down in drying racks until dry.

5.1.8 Store clean, dry glassware in dust-free cabinets or drawers.

5.1.9 Change acid baths (1:9 nitric acid) for fleakers every Monday, Wednesday, and Friday.

5.2 Bottles for Hg Analysis — Cleaning Procedures.

5.2.1 Rinse bottles 2 times with tap water.

5.2.2 Rinse off Sharpie labels with methanol.

5.2.3 Remove any residue from bottles with brush and detergent. If a brown residue persists, rinse with hydrox solution.

Hydrox Solution:

- 60 g sodium chloride
- 60 g hydroxylamine sulfate
- Dilute to 500 ml with deionized water.
- Mix well.

5.2.4 Place bottles into mercury washtubs containing 10% nitric acid and 200ml hydrox solution per tub. Soak 30 minutes or more.

Nitric Acid:

- Change acid water every Tuesday morning and every Friday morning. Be certain the washtub is cleaned before adding fresh, deionized water and acid.

5.2.5 Rinse 3 times with deionized water.

5.2.6 Place upside down in drying baskets *without* paper towels.

5.3 HG Digestion Lids

5.3.1 After soaking, scrub lids with a brush and detergent.

5.3.2 Rinse 3 times with tap water, then 3 times with deionized water.

5.3.3 Place lids into plastic tray.

5.3.4 Fill tray with 10% nitric acid and 100ml hydroxylamine solution (see Section 5.2.4 above). Soak 30 minutes or more.

5.3.5 Rinse with deionized water.

5.3.6 Place into drying tray with paper towels.

5.4 Organic Analysis Glassware Preparation

5.4.1 Although procedures for glassware vary somewhat according to the type of apparatus involved, the protocol for most apparatus involves thorough washing, solvent rinsing and high temperature oxidation. This procedure has been proven extremely effective for elimination of contamination in trace level organic analyses.

5.4.2 Glassware preparation procedure for non-volumetric glassware:

5.4.2.1 Rinse 1-2x with methanol to help remove residue.

5.4.2.2 Thoroughly wash in DI water and Alconox to remove particulate matter and gross contamination.

5.4.2.3 Rinse extensively with DI water.

5.4.2.4 Methanol rinse.

5.4.2.5 Place in oven at 200°C for 2 hours.

5.4.3 For preparation of volumetric glassware:

5.4.3.1 Rinse 1-2x with methanol to help remove residue.

5.4.3.2 Thoroughly wash in DI water and Alconox to remove particulate matter and gross contamination.

5.4.3.3 Rinse extensively with DI water.

5.4.3.4 Submerge in concentrated "Nochromic"/sulfuric acid batch for 2-4 hours and then rinse 5x with DI water (optional).

5.4.3.5 Rinse 2x with methanol.

5.4.3.6 Air Dry

5.4.3.7 Cap with aluminum foil.

5.4.3.8 Rinse 3x with solvent of choice before using.

5.5 Segregation of Potentially Contaminated Glassware

5.5.1 When a sample is suspected of containing a high contaminant level, disposable glassware will be used as much as possible and discarded after use. Any non-disposable glassware that is used will be washed and prepared separately using the SOP for glassware preparation.

5.5.2 Glassware suspected of gross contamination will be rinsed with methylene chloride three times, the rinsate concentrated to a final volume of 0.5ml, and analyzed by chromatographic techniques to check for residual contamination. Glassware checks exhibiting detectable peaks will be discarded.

5.5.3 The suspected contaminated glassware will be placed in a nonchromix solution overnight, after which it is rinsed thoroughly with tap water and detergent, followed by liberal rinsings of DI water, and finally rinsed with Methanol and dried for 2 hours.

6.0 RECORDS. N/A

7.0 REFERENCES. N/A

8.0 ATTACHMENTS. N/A

9.0 DEFINITIONS. N/A

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GA-124/-127/-128

The Standard Operating Procedures (SOPs) contained herein are adopted by Pace Analytical Services.

These SOPs will be followed until superceded by Pace Analytical Services SOPs.

Signature:	Signature:	Signature:
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SOUTHWEST LABORATORY OF OKLAHOMA, INC. *and*
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Standard Operating Procedure

LIMS For Sample Receiving

Document No.: SWL-GA-124

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LIMS FOR SAMPLE RECEIVING

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1.0 PURPOSE AND APPLICATION

LIMS is used as the Sample Custodians main tool for managing sample information. This Standard Operating Procedure is used to give a step by step process for entering sample information into the LIMS system.

2.0 RESPONSIBILITIES

2.1 It is the responsibility of the Project Officer to provide the Sample Custodian with an ARF (Analytical Request Form) for each episode.

2.2 It is the responsibility of the Sample Custodian to enter accurate sample information and to quality check all information between the COC (Chain of Custody), ARF, and logged paperwork.

3.0 EQUIPMENT

3.1 LIMS Access

3.2 HP Laser Jet Printer or equivalent

3.3 Eltron direct thermal printer or equivalent

4.0 PRECAUTIONS

See Standard Operating Procedure "*Laboratory Safety Plan*", SWL-GA-111.

5.0 PROCEDURE

5.1 Log On

5.1.1 To log onto LIMS, click on the LIMS icon on the desktop.

5.1.2 You must provide user name and password.

5.2 Sample Receiving Screen

The sample receiving screen gives you options for *Samples, Reports,* and *Help.*

5.3 Samples Key

The "Samples" menu gives you options for *New, Modify, Disposal, Barrel ID's, ARF Login, ARF Login by Ref., SDG Assignment, Rapid Turn Notify,* and *Exit.*

5.4 Episode Login

There are two ways to log new episodes: *ARF Login,* by *reference,* and *New Login.*

5.4.1 ARF Login by Ref

5.4.1.1 Choose *ARF Login by Ref* from the *Samples Menu.*

5.4.1.2 Provide the *ARF Ref #, Located on the upper right corner of the ARF.*

5.4.1.3 The computer will now retrieve information provided on the *ARF*, by the Project Officer, and leave you at the *Login Sheet* screen.

5.4.1.4 On the *Login Sheet* tab you must provide the following:

- *Received by:* Name, Date, and Time
- *Logged by:* Name, Date, and Time
- *Custody Seal:* Status and Numbers (Enter "N/A" if no seal numbers are present on cooler; enter "No Seals" if no custody seals were present.)
- *Airbill:* Status and Number(s) (Enter "*Walk In*" for all customer drop-offs)
- You must click all of the applicable on *COC Records* (Present, Sealed in Plastic, Taped to Lid, and Properly Filled Out)
- You must click on the following items that pertain to this episode (i.e. Traffic Report…, Do COC…, Cooler Temp..., Rad Screen…, PO Called)
- *Sample Tags:* One of the following must be chosen (Absent, On COC, Not on COC)
- *Sample Condition:* Status (Click on all of the following that pertains to the episode)
- *Cooler Condition:* Enough Ice (click if cooler temp is below 6°C), choose the appropriate type of ice, and packing material
- *Cooler ID / Temperature:* This is for EPA use only; use the following format for cooler id's (i.e. 10/01/99-1, 10/01/99-2)

5.4.1.5 On the *Episode* tab you must provide the following:

- All of the following fields should be filled by use of the *ARF Login* step, Client, Project, and Lab ID.
- Site, P.O. Number, Comment, and Case No. can be found on the ARF (EPA Case #'s are listed on Traffic Report)
- *Report Print Options* and *Log Sheet Print Options* are set to default and should only need the Login Sheet Option changed in the event of an EPA episode.

5.4.1.6 On the *Samples* tab you must provide the following:

- You must choose between Invoice and OnHold (***Invoice*** is the default setting and *OnHold* is to be chosen when designated by the Project Officer)
- Client ID: This is filled during the *ARF Login* step.
- Type: This is filled during the *ARF Login* step. (i.e. N1, MS1, SD1, DU1)
- Matrix: This is filled during the *ARF Login* step.
- Sampled: This must contain the date and time sampled, taken from the COC/Traffic Report.
- Containers: Must have total number of containers for sample ID.

- Location, Depth From ~ To: This will be filled with the information contained on the COC, if provided.
- SDG: This is filled during the *ARF Login* step.
- Short ID: This will be filled with information contained on the COC, if provided.
- Descr.: This will be filled with information contained on the COC, if provided.
- Submitter: This is filled during the *ARF Login* step.
- Field Lot #: This will be filled with information contained on the COC, if provided.
- Cooler ID: This will be in the following format: mm/dd/yy-#, (i.e. 10/01/99-1, 10/01/99-2). Assign each cooler ID to the samples that arrived in those coolers.
- Tags: This is used to list EPA control tag numbers.
- Shipping condition: Provide the cooler temp in this field. (All temps are in Celsius, designate this by the use of a lower case "c" after the numeric temperature.
- Sample Fractions: Will be assigned by the program on a basis of test codes.
- ACID Base Lot #: Fill if information is provided.
- Holding Time: Defaults to standard of ninety days.
- Storage: *Not yet assigned*
- Shelf: *Not yet assigned*
- pH Check: Check when containers in the sample ID have preservatives used and a pH check has been performed.
- Copy Sample & Container Data Forward: This box defaults with a check and will carry all information to the next sample ID.

5.4.1.7 On the Analysis tab the following fields must be filled:

- Test: This is filled during the ARF Login step.
- Inv. Item: This is filled during the ARF Login step.
- RptList#: This is filled during the ARF Login step.
- Description: This is the default nomenclature for the test code.
- Cont: Container letter designations must be assigned to the correct test codes.
- Pri: Is assigned (as follows) during the ARF Login step, according to the due date listed on the ARF.

  — 3 = 30 days
  — 4 = 21 days
  — 5 = 14 days
  — 6 = 7 days
  — 7 = 5 days
  — 8 = 2 days
  — 9 = 1 day

- Due: The program automatically calculates this date. The date can be changed for dates that do not fall in to the standard turnaround times listed above.

5.4.2 New Login

During new login, all procedures from the *ARF Login* are followed, however you must provide all information that previously would have been pulled over on the ARF Login step.

5.5 Documentation

5.5.1 After all information is entered on the episode, you must go to the Episode screen and click on the *DONE* button.

5.5.2 The following documents should print at completion:

- Work Sheet
- DC-1 (Cooler Receipt)
- Internal COC
- Labels

5.6 Reports

5.6.1 The following options can be used to reprint forms.

5.6.1.1 *Work Sheet*: Must provide episode number

5.6.1.2 *Labels:* Must provide episode number or episode and sample number (i.e. "41976.01" will give you all containers for ".01")

5.6.1.3 *Cooler Receipt — DC1*: Must provide episode number and choose EPA / Non-EPA.

5.6.1.4 *Chain of Custody*: Must provide episode number.

6.0 RECORDS

Not applicable.

7.0 REFERENCES

Not applicable.

8.0 DEFINITIONS

COC: .....Chain of Custody. Field document used to document and relinquish custody of sample from field.

Internal COC:.....Internal Chain of Custody. Document to record custody of samples through the laboratory.

DC-1:.....Document Control-Form #1

ARF:.....Analytical Request Form. A form completed by the Project Officer and used by the Sample Custodian, to detail what analyses is required for each sample.

LIMS:.....Laboratory Information Management System.

Traffic Report: .....Chain of Custody used by the EPA.

9.0 ATTACHMENTS

Not applicable.

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Standard Operating Procedure

Preventive Maintenance

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PREVENTIVE MAINTENANCE

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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.

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*
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Standard Operating Procedure

Maintenance of "Zone" Log Books

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MAINTENANCE OF "ZONE" LOG BOOKS

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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) — Diana Hoke/Ann Wade
- OL/GC Pesticides (OP) — Blaine Bolding/Imelda Williams
- RMCA (RD) — Kert Surface/David Hayes
- Volatile GC/MS (OV) — Sam Alexander/Janet Williamson
- Organic Extractions (OE) — Desmond Foster/Chris Derouen
- Sample Receiving (SR) —Jason Spriggs/Debra Klapperich
- Inorganics/Metals (IN) —Susan Turner/Melissa Self
- Air/Dioxin (OA/OD) — Jayant Shringarpure/Barry Hall/Bryon Landtroop
- GC-VOA (OG) —Bryon Landtroop/Sam Alexander

The logs will be maintained by designated employees and reviewed by the above supervisors. An audit check will be done on all new zone logbooks 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/Resistivity Checks" 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" shall be maintained in a bound zone logbook in each laboratory area, which requires the monitoring of hot plate temperatures.

7.0 REFERENCES

Not applicable.

8.0 DEFINITION

- Zone = A defined area of the laboratory.

9.0 ATTACHMENTS

9.1 Attachment 1 — Refrigerator Temperature Record, GA128F1.

9.2 Attachment 2 — Freezer Temperature Record, GA128F2.

9.3 Attachment 3 — Oven Temperature Record, GA128F3.

9.4 Attachment 4 — D.I. Water Conductivity/Resistivity Checks, GA128F4.

9.5 Attachment 5 — Daily Balance Checks, GA128F5.

9.6 Attachment 6 — Fume Hood Daily Flow Check, GA128F6.

9.7 Attachment 7 — Equalization Tank Weekly pH Check, GA128F7.

9.8 Attachment 8 — Water Bath Monitoring Record, GA128F8.

9.9 Attachment 9 — Sonicator, GA128F9.

9.10 Attachment 10 — Sample Receipt Thermometer Calibration Check, GA128F10.

9.11 Attachment 11 — pH Meter Calibration Log

9.12 Attachment 12 – Hot Plate Monitoring Log

ATTACHMENT 1
Refrigerator Temperature Record

Refrigerator Temperature Record, SWL-GA-128

GENERAL ADMINISTRATIVE

SOP REV. # 4.0 09/05/01 FILE ID: GA128F1.doc Book ID: GA-128-ZoneRef-___

REFRIG # : _____
MONTH/YEAR: _____
THERM. ID: _____

CONTROL LIMITS: __2.0___°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 REV. # 4.0 09/05/01 FILE ID: GA128F2.doc Book ID: GA-128-ZoneFreez-___

FREEZER #: _____
MONTH/YEAR: _____
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

SOP REV. # 4.0 09/05/01 FILE ID: GA128F3.doc Book ID: GA-128-ZoneOven-___

GENER
ADMINISTRATI

OVEN #: _____
MONTH/YEAR: _____
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

ATTACHMENT 4
D. I. Water Conductivity/Resistivity Checks

D.I. Water Conductivity/Resistivity Checks, SWL-GA-128

GENERA ADMINISTRATIVI

SOP Rev.5.0 10/18/02 File ID: GA128F4.doc Book ID: GA-128-ZoneDI-___

MONTH/YEAR: _____

CONDUCTIVITY ACCEPTANCE: <1.0 UMHOS/CM
RESISTIVITY ACCEPTANCE: >16.67 MΩ - cm

Date	Lab Area	Conductivity umhos/cm	Indicator Light	Analyst	Resistivity MΩ - cm	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				
13			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 Checks, SWL-GA-128

SOP Rev.4.0 09/05/01 File ID: GA128F5 Book ID: GA-128-ZoneBaln- _____

MONTH/YEAR: _____

WT. SET USED: _____

(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											
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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

SOP Rev. 5.0 01/30/03 File ID: GA128F6 Book ID: GA-128-ZoneFume-3

GENERA
ADMINISTRATIVI

MONTH/YEAR: _____

ACCEPTANCE: 75 – 200 FPM at Sash Mark

DATE	LAB	FUME HOOD NUMBER	FLOW CHECK (FULL FACE)	FLOW CHECK (sash mark)	ANALYST
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					
13					
14					
15					
16					
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29					
30					
31					

REPAIR SERVICE: Tobey's Heating & A/C • Ken Toby • 664-1166

TYPIST: LLR

ATTACHMENT 7
Equalization Tank Weekly pH Check

Equalization Tank Weekly pH Check, SWL-GA-128

GENERAL ADMINISTRATIVE

SOP Rev. 5.0 01/30/03 File ID: GA128F7 Book ID: GA-128-ZoneTank-3

MONTH/YEAR: _____

ACCEPTANCE: 3 — 11

DATE	LAB AREA	pH READING	ANALYST	COMMENTS
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				
11				
12				
13				
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26				
27				
28				
29				
30				
31				

TYPIST: LLR

ATTACHMENT 8
Water Bath Monitoring Record

Water Bath Monitoring Record, SWL-GA-128

GENERAL ADMINISTRATIVE

SOP REV. # 4.0 09/05/01 FILE ID: GA128F8.doc Book ID: GA-128-ZoneBath-___

MONTH/YEAR: _____

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

SOP REV. # 5.0 01/30/03 FILE ID: GA128F9.doc Book ID: GA-128-ZoneSoni-___

MONTH/YEAR: _____

CONTROL LIMITS: 17.5 (±3) DIV

DATE	TIME	ANALYST	WITHIN LIMITS	ACTUAL READING	SONICATOR #	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

MONTH/YEAR: _____

CONTROL LIMITS: ± 2 Degrees C.

DATE	S.R. I.D.	Therm. Reading	NIST Therm. I.D.	Therm. Reading	S.R. Bias	Therm. Analyst	Corrective Action (If Needed)
1							
2							
3							
4							
5							
6							
7							
8							
9							
10							
11							
12							
13							
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26							
27							
28							
29							
30							
31							

ATTACHMENT 11
pH Meter Calibration Log

pH Meter Calibration Log, SWL-GA-128

SOP REV. # 4.0 09/05/01 FILE ID: GA128F11.doc Book ID: GA-128-pHMeter-___

MONTH/YEAR: _____

CONTROL LIMITS: Slope 95-102

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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19						
20						
21						
22						
23						
24						
25						
26						
27						
28						
29						
30						
31						

ATTACHMENT 12
Hot Plate Monitoring Log

Hot Plate Monitoring Log, SWL-GA-128

GENERAL ADMINISTRATIVE

SOP REV. # 4.0 09/05/01 FILE ID: GA128F12.doc Book ID: GA-128-ZONEHP-___

FOR THE MONTH OF : _____

1	2	3	4
5	6	7	8
9	10	11	12

Acceptance range: ILM04.1: 92°C to 95°C SW846: 90°C to 95°C

	Position # 1		Position # 2		Position # 3		Position # 4		
Day	Spot	Temp	Spot	Temp	Spot	Temp	Spot	Temp	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									

Temperatures are in degrees Celsius (°C)

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GA-133 / -135 / -139 / -140

The Standard Operating Procedures (SOPs) contained herein are adopted by Pace Analytical Services.

These SOPs will be followed until superceded by Pace Analytical Services SOPs.

Signature:	Signature:	Signature:
Chuck Hoover	Roger C. Splinter	Dave Neal
Quality Assurance Officer	Dir. of Quality, Safety, Training	General Manager
Pace Analytical Services, Inc.	Pace Analytical Services, Inc.	Pace Analytical Services, Inc.
Broken Arrow, Oklahoma	Minneaplolis, MN 55414	Lenexa, KS

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

TM

Standard Operating Procedure

Usage of Blanks and Backup Blanks

Document No.: SWL-GA-133

Rev No. / Date: Rev. 2.1 — 09/15/99

OFFICIAL COPY RED STAMP INDICATES ORIGINAL

APPROVALS	DATES
Harry M. Borg Procedure Prepared By	9-15-99 Date
Harold L. Markle Laboratory Manager or Director	09/15/99 Date
Chuck Hoover Laboratory Safety Officer	9/15/99 Date
Chuck Hoover Laboratory QA/QC Officer	9/15/99 Date

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

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

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USAGE OF BLANKS AND BACKUP BLANKS

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8.0 ATTACHMENTS 2

Attachment 1: Back-up Blanks Usage Form 3

9.0 DEFINITIONS 2

1.0 PURPOSE & APPLICATION— The purpose of this standard operating procedure is to provide understanding and guidance on the implementation and proper usage of back-up blanks associated with extraction and analyses of organic compounds.

2.0 RESPONSIBILITIES — It is the responsibility of the extraction chemist to set up blanks per extraction batch following this Standard Operating Procedure.

3.0 EQUIPMENT — Refer to individual extraction and/or analytical standard operating procedure.

4.0 PRECAUTIONS — See individual analytical SOP.

5.0 PROCEDURE

5.1 Blank nomenclature — Blanks will have the naming format of: TTMMDDX#

Where: TT = Type: (BL = Extraction Blank
BB = Back-Up Blank)
MM = Month
DD = Day
X = Matrix: A = Air
AG = Agriculture
E = Emulsion
O = Other
P = Petroleum
S = Soil (including SD=Solid, SL=Sludge)
W = Water
WI = Wipe

 # = Unique alphabetic identifier (A-Z) (one per prep batch).

Example: The first extraction batch of waters performed on 1/1/95 would have blank names of BL010195WA for the extraction blank and BB010195WA for the back-up blank. The first extraction batch of soils performed on 1/1/95 would have blank names of BL010195SA for the extraction blank and BB010195SA for the back-up blank. The second batch of either matrix for this day would have the same names except that the A would be changed to a B in the final character of the name. Using this convention, there is a possibility of 26 extraction batches per matrix per day.

5.2 Blank Usage

5.2.1 The extraction blank (BLmmddx#) will be the primary blank used for analysis. This blank must be analyzed. The back-up blank (BBmmddx#) is to be used <u>only</u> when <u>certain</u> problems associated with the primary extraction blank have occurred. When a back-up blank is used, a back-up blank usage form (FORM 1) must be filled out by the analyst and signed by supervisor. This form must accompany any extra data associated with the samples. A copy of this form must be submitted to supervisor and retained on file.

5.2.2 The following is a list of problems in which a back-up blank my be used:

5.2.2.1 Primary blank was lost during the extraction process or extract container either broke or went dry before analysis.

5.2.2.2 Primary blank had low surrogate recoveries, and associated samples did not indicate the same low surrogate recoveries. Any samples having the same low surrogate recoveries would be first re-analyzed and if necessary re-extracted. If re-extracts are out side of hold time, then report both primary blank, back-up blank, and both original extract analyses and re-extract analyses. Note problem in case narrative.

5.2.2.3 Primary blank had internal standards outside of QC limits and associated samples did not indicate the same problem. Any samples having the same internal standard area problems would be first re-analyzed and if necessary re-extracted. If re-extracts are out side of hold time, then report both primary blank, back-up blank, and both original extract analyses and re-extract analyses. Note problem in case narrative.

5.2.2.4 Primary blank contains contamination of target analytes above the reporting limit (for phthalates 5 times the reporting limit) and associated samples did not indicate the same problem. Any samples having the same compounds above the reporting limits, would first be re-analyzed and if necessary re-extracted to verify that compounds were not laboratory contamination. Exceptions of re-extraction would be when compounds found in sample are above 10 times the amount found in the blank. If re-extracts are out side of hold time, then report both primary blank, back-up blank, and both original extract analyses and re-extract analyses. Note problem in case narrative.

5.2.2.5 Primary blank contains trace contamination of target analytes and the associated samples did not indicate the same problem.

6.0 RECORDS — Records will be maintained in the extraction log as per the analytical or extraction SOP.

7.0 REFERENCES — N/A

8.0 ATTACHMENTS — Back-up Blanks Usage Form

9.0 DEFINITIONS — N/A

ATTACHMENT 1
Back-up Blank Usage Form

BACK-UP BLANK USAGE FORM

SWL-GA-133, REV. 2.0 — 07/31/97, SWL-GA-133-BKUPBLK

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:_____

_____

SOUTHWEST LABORATORY OF OKLAHOMA, INC. and
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ating Procedure

Standard C

gin Review

Sample

.: SWL-GA-135

ev. 2.0 — 12/20/01

Docume

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CIAL
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DATES

1-26-02
Date

1/28/02
Date

1/28/02
Date

1/28/02
Date

er the last signature above - QA/QC Officer)

APPROVALS

Procedure Prepared By

Laboratory Manager or Director

Laboratory Safety Officer

Laboratory QA/QC Officer

(Effective Date for is 10 calendar d

Date: QA:

TT NUS-PA Date: 10/17/03 QA: CAH

Document Status

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SAMPLE LOGIN REVIEW

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- **9.2** Acronyms 3

1.0 PURPOSE AND APPLICATION

1.1 The purpose of this procedure is to provide guidelines for sample log-in review. It will provide specific instructions to ensure the review process is performed with consistency.

1.2 This procedure applies to all samples received at Southwest Laboratory of Oklahoma, Inc. This procedure details the step by step procedures for the review of "*Cooler/Sample Log-in Sheets*" (Form-DC1 — see SWL-GA-115) and sample worksheets before the samples are released to the laboratory.

2.0 RESPONSIBILITIES

2.1 The Sample Custodians are responsible for the review of log-ins performed by their peers.

2.2 The Laboratory Quality Assurance Officer is responsible for the incorporation of quality assurance program requirements appropriate to the review of sample log-in.

2.3 The Project Officers are responsible for the peer review of sample log-ins when only one sample custodian is available or when the workload precludes the sample custodian peer review.

2.4 The Program Managers or their designees are responsible for assuring that all sample worksheets received in their departments have been reviewed before starting analysis.

3.0 EQUIPMENT

Not Applicable.

4.0 PRECAUTIONS

Not Applicable

5.0 PROCEDURE

5.1 All samples will be logged in according to procedures outlined in SOP SWL-GA-115, *"Sample Custodian"*. Prior to releasing the samples to the laboratory, the sample custodian must generate all necessary paperwork and then submit it to a department peer for a detailed peer review. All reviews must be performed the day that the samples are logged in. All paperwork must be scanned/copied and distributed to appropriate laboratory by end of day on samples with turn-around time longer than 7 days. Samples with turn-around times of 7 days or less must be reviewed, scanned/copied and distributed at the time of login.

5.2 The reviewer will check the following items:

5.2.1 All paperwork is present including the "*Sample Work Sheet,*" "*Chain-of-Custody*" (COC), "*Traffic Report*" (TR), "*Cooler Receipt/Sample Log-in Sheet,*" and the "*Analytical Request Form*" (ARF).

5.2.2 That the client sample ID on the "*COC/TR*" matches those on the "*Cooler Receipt/Sample Log-in Sheet*" (Form-DC1 — see SWL-GA-115) form, the "*Analytical Request Form*" and the "*Sample Worksheet*", and the sample bottle label(s).

5.2.3 All items on "*Cooler Receipt/Sample Log-in Sheet,*" *(Form-DC1, see SWL-GA-115)* are filled in correctly, including:

- Episode Number matches "*Sample Worksheet*".
- Project ID matches ARF.
- SDG number matches ARF.
- Remarks section filled out.
- All sample fractions are present for each sample.
- The pH of preserved samples were checked.
- The acid/base lot # added for samples that required pH adjustment.
- Condition of Samples filled out.

5.2.4 If a facility transfer is required for samples check to assure that the "*Facility Transfer Form*" is correct.

5.2.5 Check test codes on "*Sample Worksheet*" to see if they match those on the ARF.

5.2.6 Are due dates and matrix correct on the "*Sample Worksheet*".

5.2.7 After review, the original "*Sample Worksheet*" will be stamped "Reviewed" with the reviewer's signature and date of review. The worksheets will then be scanned, copied, and distributed to the lab.

5.2.8 If Project Officers will be performing the log-in review then all original paperwork must be forwarded to them. Once they have reviewed and approved the log-in the original worksheet will be stamped "Reviewed" with reviewer's signature and the date of review. All original paperwork will then be returned to the Sample Custodian for scanning/copying and distribution to the laboratory.

6.0 RECORDS

6.1 LIMS generated "*Sample Worksheet*". A description can be found in SOP document ID SWL-GA-115.

6.2 Clients supplied "*Chain-of-Custody/Traffic Report*". A description of chain-of-custody can be found in SOP document ID SWL-GA-110.

6.3 "*Analytical Request Form*". Document Control ID "GA-131-AREQ-F".

6.4 "*Facility Transfer Form*". Document Control ID "GA134-Trans-F".

7.0 REFERENCES

7.1 Reference SWL document ID, SWL-GA-110, "*Laboratory Documentation of Sample Custody*".

7.2 Reference SWL document ID, SWL-GA-115, "*Sample Custodian*".

8.0 ATTACHMENTS

Not Applicable.

9.0 DEFINITIONS

9.1 Terminology

9.2 Acronyms

SOP: ..... Standard Operating Procedures are official documents, which define policy and describe the procedures involved in the operation of the laboratory.

COC: ..... Chain-of Custody is an evidentiary document, identifying anyone having had custody of the samples listed. Information contained on a COC also includes (but is not limited to); client sample ID's date/time of sample collection, analytical test to be performed, matrix, etc.

LIMS: ..... Laboratory Information Management System is a networked database, which drives the production of analytical results, reports, and invoicing for the laboratory. The process begins with the logging in of a sample.

TR: ..... Traffic Report is an evidentiary document, identifying anyone having had custody of the samples listed. Information contained on a TR also includes (but is not limited to); client sample ID's date/time of sample collection, analytical test to be performed, matrix, etc.

SOUTHWEST LABORATORY OF OKLAHOMA, INC. *and*
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Standard Operating Procedure

Maintenance and Management of the LIMS System

Document No.: SWL-GA-139

Rev No. / Date: Rev. No.: 3.0 — 03/13/03

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APPROVALS	DATES
Mark Smith Procedure Prepared By	3-14-03 Date
Robert W. Marr Laboratory Manager or Director	3-25-03 Date
Chuck Jones Laboratory Safety Officer	3/27/03 Date
Chuck Jones Laboratory QA/QC Officer	3/27/03 Date

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

Document Status

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MAINTENANCE AND MANAGEMENT OF THE LIMS SYSTEM

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1.0 PURPOSE AND APPLICATION

1.1 The purpose of this standard operating procedure is to provide guidelines for the maintenance and management of the Laboratory's information management system. Currently, Southwest Laboratory of Oklahoma, Inc.'s (SWL) LIMS system utilizes a Windows NT based network with SQL Server as the relation database management tool.

1.2 Although application of the LIMS involves almost every aspect of SWL's sample processing (see SWL-GA-102,) the application of this document specifically addresses those areas of responsibility performed by SWL's System Administrator. The following procedures shall provide guidelines for; 1) nightly backups on all databases and servers connected to the LIMS; 2) monitoring the System for error messages; 3) monitoring free space on both the file system and the database; 4) removal of temp files; 5) archival of data; and 6) adding new users and passwords to LIMS.

2.0 RESPONSIBILITIES

2.1 The LIMS Administrator or their designee is responsible for the tasks included in this Standard Operating Procedure.

2.2 The Information Systems Manager has oversight responsibilities for all tasks detailed in this procedural document.

2.3 The Laboratory Director is responsible for seeing that Computer Services has sufficient hardware and software to accomplish the tasks outlined in this SOP.

3.0 EQUIPMENT

3.1 Hardware — LIMS and Windows NT-based Servers are Pentium PC's with Intel based processors and are located throughout the lab.

3.1.1 Exabyte Mammoth 2 Tape Drive – Used for making backups of LIMS Database and NT servers.

3.2 To be fully operational, the LIMS currently requires the commercial programs (software) briefly described below. Please refer to Southwest Laboratory's procedural document.

3.2.1 Windows NT — operating system with a TCP/IP network.

3.2.2 SQL Server — relational database management tool.

3.2.3 Veritas Backup Exec (version 8.5) – Used for controlling tape backup and restore functions.

4.0 PRECAUTIONS

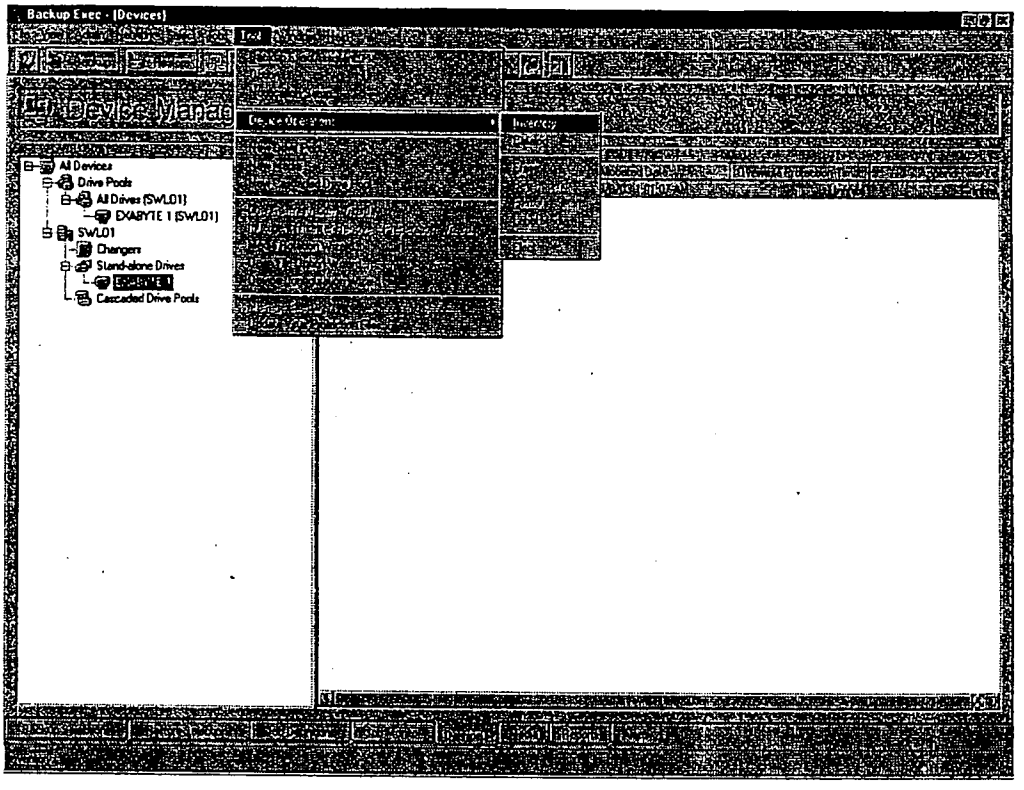
Not applicable.

5.0 PROCEDURE

5.1 The verification and maintenance of backups on the laboratory's LIMS and network systems are performed system wide and on departmental databases on a daily and weekly basis. For step by step instruction, see SOP SWL-GA-138, "*LIMS Backup Procedures*."

5.1.1 NT Server Backup and Restoration Functions - All NT tape backup and restoration functions are controlled through the Veritas Backup Exec program. The program is accessed through Start | Programs | VERITAS Backup Exec.

5.1.1.1 Device Functions – Before performing a backup or restore, the correct tape must be in the drive. The tape must be inventoried, and if a restoration is being performed, cataloged. These functions are accessed through the "Devices" tab or the Tools | Device Options menu (See screen print below). If accessed through the "Devices" tab, right click the device name to show the pop-up menu.

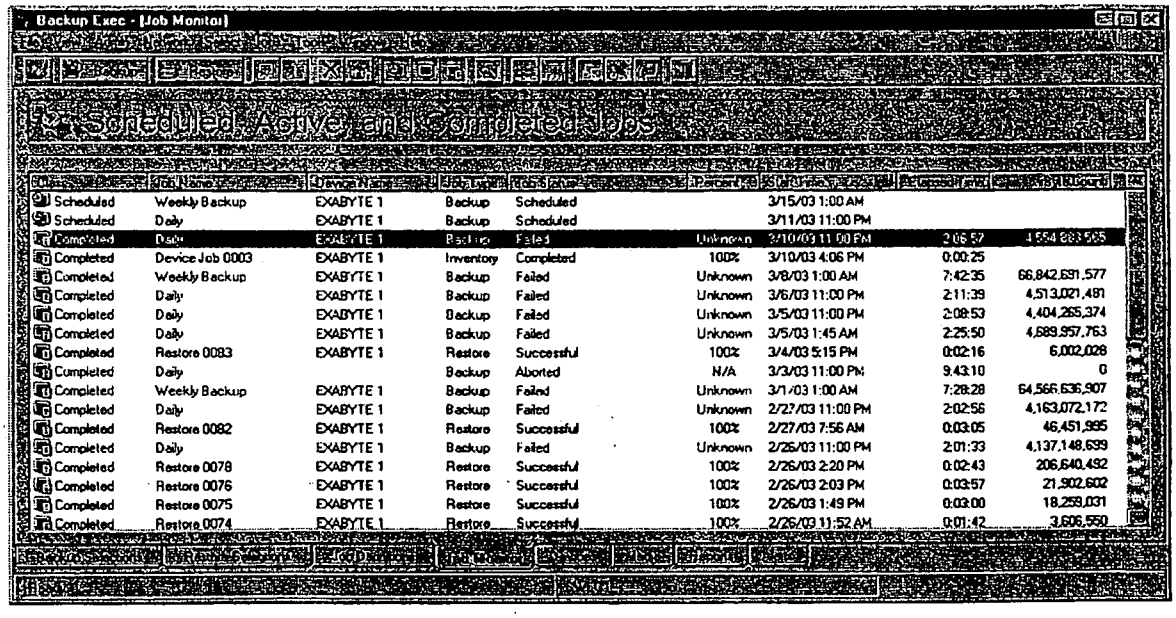


- Eject – Rewind and eject the current tape in the drive.
- Inventory – Determine the tape id of the current tape in the drive.
- Catalog – Create a directory of the tape.

5.1.1.2 Scheduled Backups – The NT servers have both daily and weekly scheduled backups. The daily backup stores all non-operating system files for all servers that have been changed that day. These backups occur each weekday evening (except Friday) around 11 PM. Multiple daily backups may be stored on a single tape. The tape may be reused until it reaches capacity. The weekly backup stores all non-operating

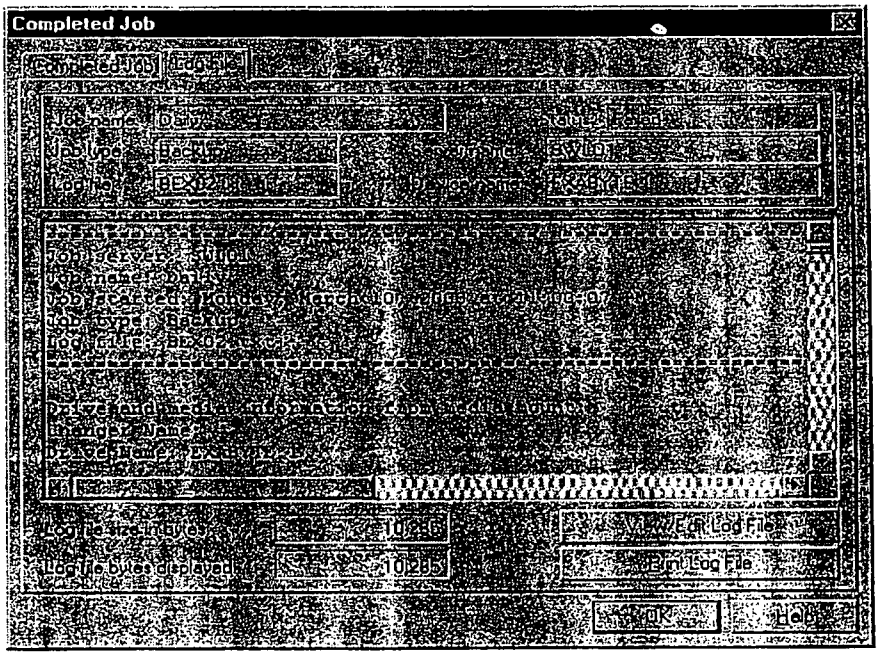
system files for all servers. This backup occurs on Saturdays at 1 AM. The scheduled jobs may be viewed through the "Job Monitor" tab (See below). A new blank tape is used for each weekly backup.

The status of the previous backup should be check the following business day. If the status shows as failed in the "Job Monitor," check the backup log. This is done by double clicking the entry in the "Job Monitor" screen. The log will be shown in a separate window (see below). Depending on the reason for the failure, the backup may need to be redone.

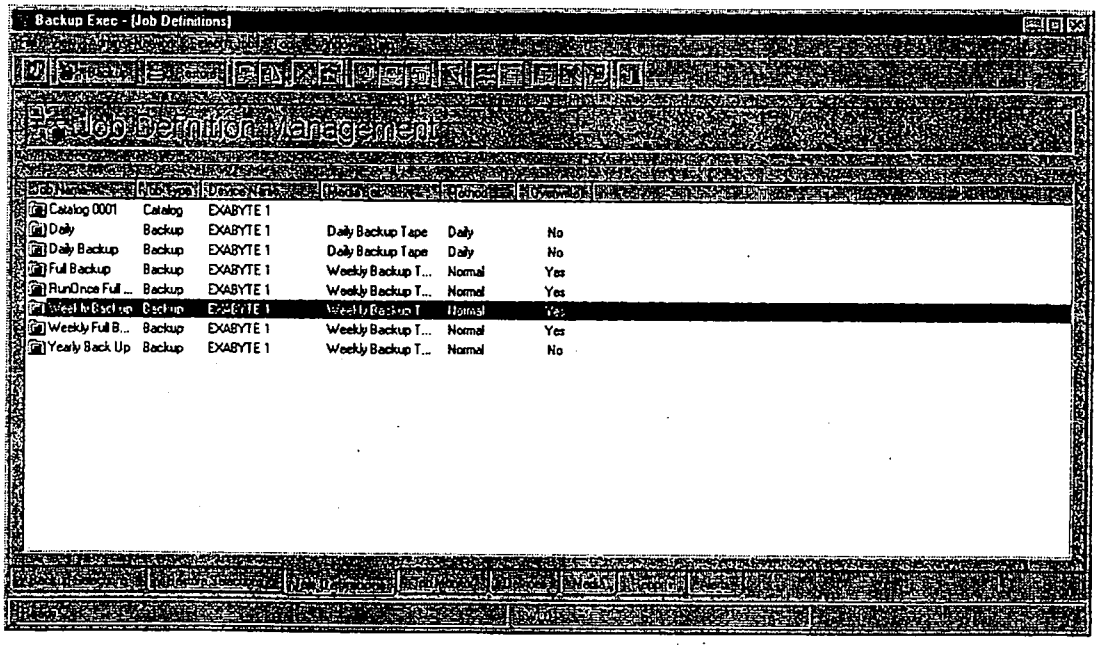


Scheduled	Weekly Backup	EXABYTE 1	Backup	Scheduled		3/15/03 1:00 AM		
Scheduled	Daily	EXABYTE 1	Backup	Scheduled		3/11/03 11:00 PM		
Completed	Daily	EXABYTE 1	Backup	Failed	Unknown	3/10/03 11:00 PM	2:06:57	4,554,888,985
Completed	Device Job 0003	EXABYTE 1	Inventory	Completed	100%	3/10/03 4:06 PM	0:00:25	
Completed	Weekly Backup	EXABYTE 1	Backup	Failed	Unknown	3/8/03 1:00 AM	7:42:35	66,842,631,577
Completed	Daily	EXABYTE 1	Backup	Failed	Unknown	3/6/03 11:00 PM	2:11:39	4,513,021,481
Completed	Daily	EXABYTE 1	Backup	Failed	Unknown	3/5/03 11:00 PM	2:08:53	4,404,265,374
Completed	Daily	EXABYTE 1	Backup	Failed	Unknown	3/5/03 1:45 AM	2:25:50	4,689,957,763
Completed	Restore 0083	EXABYTE 1	Restore	Successful	100%	3/4/03 5:15 PM	0:02:16	6,002,028
Completed	Daily		Backup	Aborted	N/A	3/3/03 11:00 PM	9:43:10	0
Completed	Weekly Backup	EXABYTE 1	Backup	Failed	Unknown	3/1/03 1:00 AM	7:28:28	64,566,636,907
Completed	Daily	EXABYTE 1	Backup	Failed	Unknown	2/27/03 11:00 PM	2:02:56	4,163,072,172
Completed	Restore 0082	EXABYTE 1	Restore	Successful	100%	2/27/03 7:56 AM	0:03:05	46,451,995
Completed	Daily	EXABYTE 1	Backup	Failed	Unknown	2/26/03 11:00 PM	2:01:33	4,137,148,639
Completed	Restore 0078	EXABYTE 1	Restore	Successful	100%	2/26/03 2:20 PM	0:02:43	206,640,492
Completed	Restore 0076	EXABYTE 1	Restore	Successful	100%	2/26/03 2:03 PM	0:03:57	21,902,602
Completed	Restore 0075	EXABYTE 1	Restore	Successful	100%	2/26/03 1:49 PM	0:03:00	18,259,031
Completed	Restore 0074	EXABYTE 1	Restore	Successful	100%	2/26/03 11:52 AM	0:01:42	3,606,550

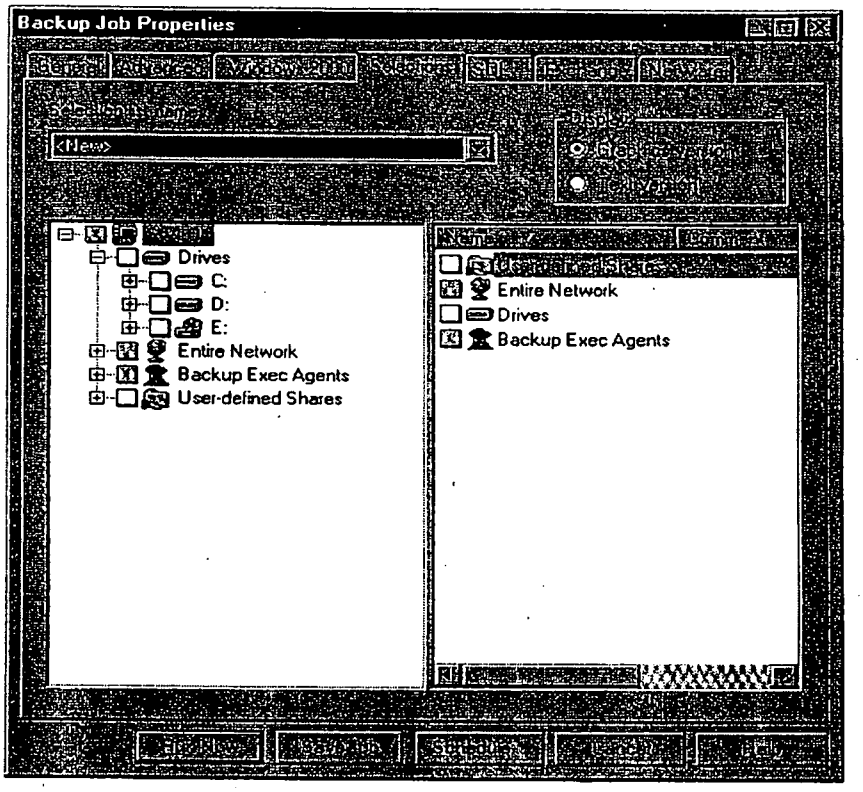
Note: A job status of failed for a backup does not mean that the backup was not successful. Backup Exec has problems verifying certain types of e-mails. When this occurs, an error is generated and the backup status is set to fail.



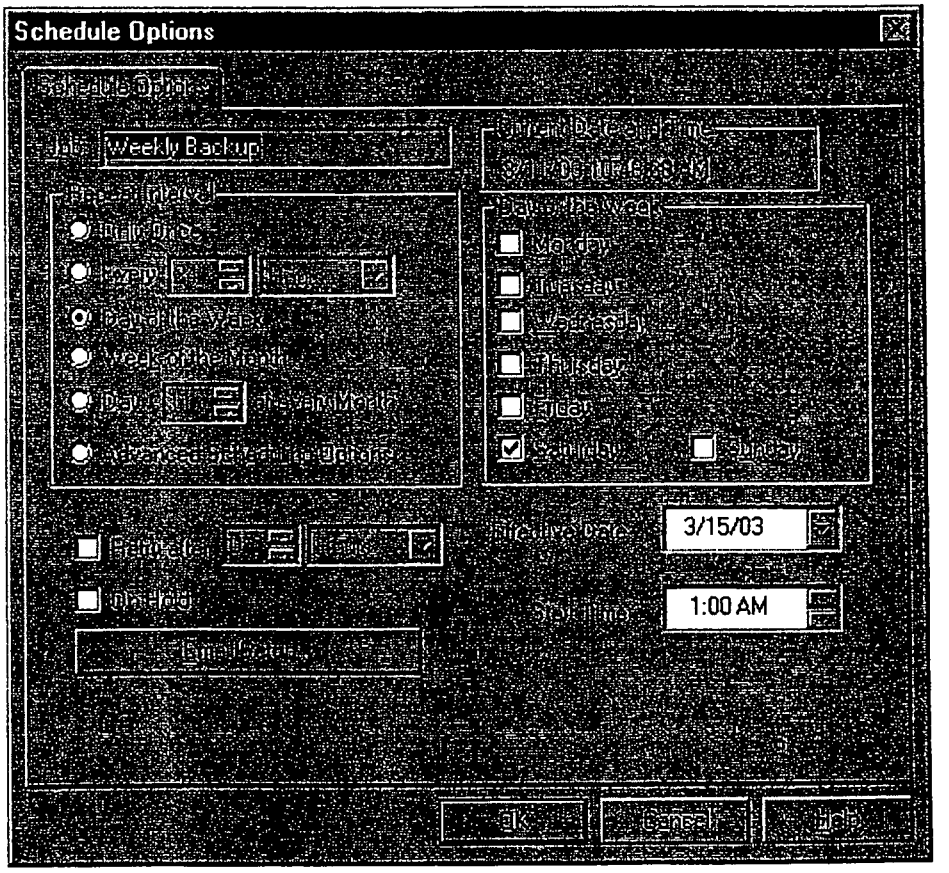
5.1.1.3 Creating a backup job – Jobs are created through the "Job Definitions" tab or the "Jobs" menu (See below). In the "Job Definitions" tab click the right mouse button and select the "New" and "Backup Job" from the pop-up menu. In the "Jobs" menu, select the "New Backup Job" option.



Select the directories and/or files from the servers in which to backup (see below).



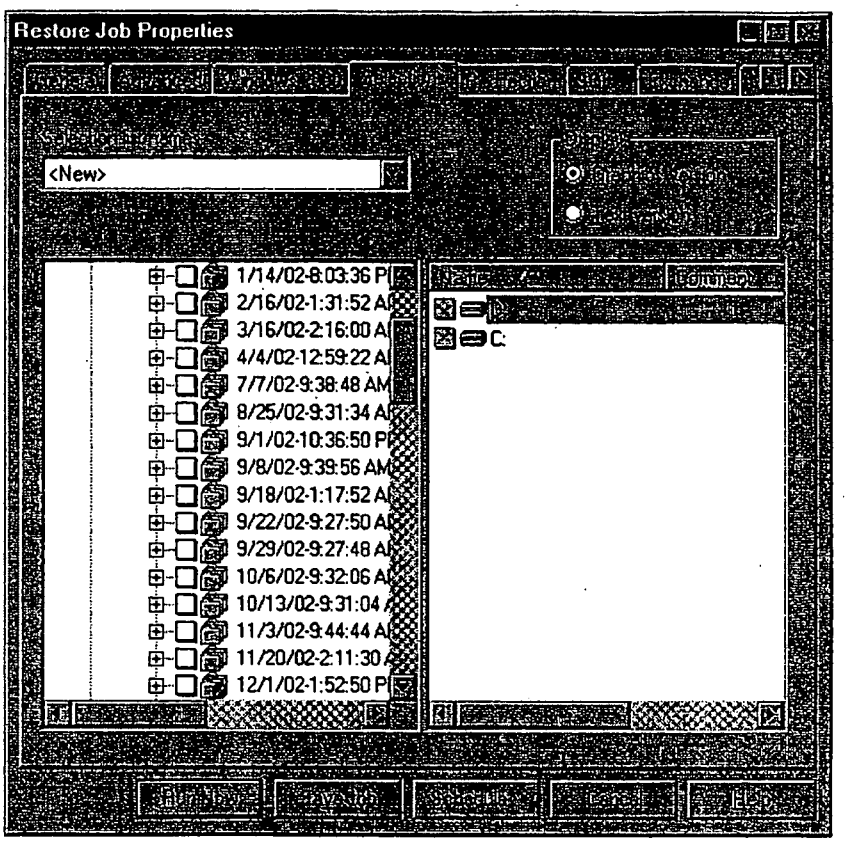
Click the "Schedule" button to setup the backup schedule. The job name may also be modified (see below). When complete click the "OK" button in the "Schedule Options." Click the "Save Job" button to store the newly created job, or the "Run Now" button to start the job immediately.



5.1.1.4 Tape Labeling – After each backup is complete, the tape should be labeled with the following information:

- Server Name on which the tape drive resides (e.g. currently the drive is connected to SWLO1).
- Type – Daily or Full
- Date of the backup
- Backup Exec Tape Number

5.1.1.5 Data Restoration – Before data can be restored from a tape, the tape must be cataloged (refer to section 5.1.1.1). Select the restore speed button located below the menu bar. Make selections of the data to restore (see below). After selection is complete select the "Run Now" or "Schedule" button. Normally, the "Run Now" option is chosen as not to interfere with the backup schedule.



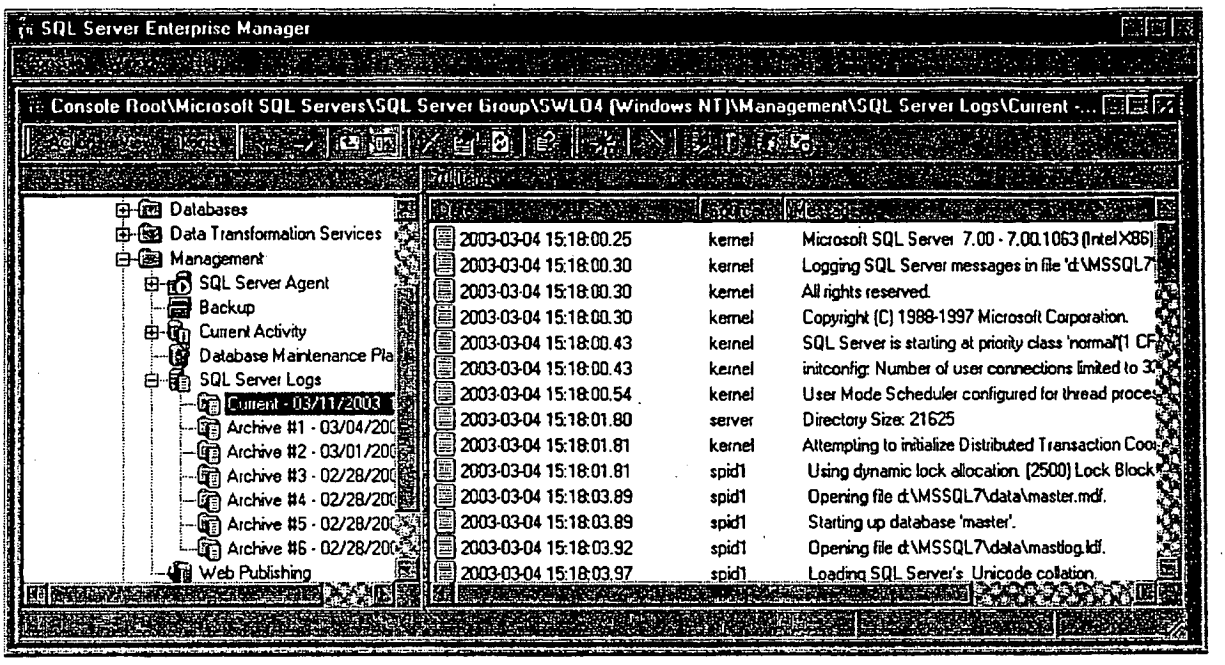
5.2 In addition to the verification and maintenance of the system/database backup responsibilities, there is a variety of miscellaneous tasks, which must be performed. Th are as follows:

5.2.1 Error Checking. In addition to the daily backup verification and maintenance routine, there are a number of error checking tasks performed at a minimum of daily or as needed.

5.2.1.1 Spooler or Printer Problems - Check the spool queues on SWLO3 for spooler or printer problems. The printer and spooler status's may be accessed through Start | Settings | Printers

5.2.1.2 System related errors – Periodically check the Event Viewer for system-related errors. Access the Event Viewer through Start | Programs | Administrative Tools (Common) | Event Viewer.

5.2.1.3 SQL Server - Periodically check the SQL Server logs for problems. The LIMS database is located on the SWLO4 server. The logs are accessed through the Enterprise Manager program. Open the program through Start | Programs | Microsoft SQL Server 7.0 | EnterPrise Manager. The LIMS database is located on the SWLO4 server. Locate the SQL Server Logs under the Management option (See below).



5.2.2 Free Space Management – Check the NT servers weekly. Inform departments when data needs to be removed.

5.2.3 Adding New Users – A request for a new user account is initiated by the department supervisor through the Computer Services Request form. Accounts are created using the User Manager program (Start | Programs | Administrative Tools (Common) | User Manager). The request form should indicate the access needed or another user who has the same access. The account should be require the user to change the login at next logon.

6.0 RECORDS

6.1 The Test Code Addition Request form (see Attachment 8 of SWL-GA-102) 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 Manager. The Manager is responsible for denying or approving, delegating and completing the Test Code Addition Request.

6.2 The Database Modification Request form (see Attachment 9 of SWL-GA-102) 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 Manager is responsible for delegating and completing the Database Modification Request after approval.

6.3 The Computer Service Request Form (see Attachment 5 of SWL-GA-102) documents the request for miscellaneous computer services. This includes Hardware Problems, Software Problems, request for change in user access, etc. The Form is completed by the Laboratory departments and submitted to the Information Systems Manager. The Information Systems Manager is responsible for delegating and completing the Computer Service Request after approval.

6.4 The Software Programming Request Form (see Attachment 6 of SWL-GA-102) documents the need for a new program, enhancement to an existing program, or a program repair. It is completed by the Laboratory departments and submitted to the Information Systems Manager. The Information Systems Manager is responsible for delegating and completing the request. It then goes back to the Requesting Individual for verification, then on to the QA Officer for validation. After completion, the LIMS Administrator will then assign a software control number to the program and file appropriately.

7.0 REFERENCES

7.1 Reference SWL-GA-102, "*Information Systems Quality Assurance Program*", Revision 1.2, Dated June 4, 1997.

7.2 Reference SWL-GA-138, "*LIMS Backup Procedures*", Revision 1.0, Dated November 5, 1997

8.0 DEFINITIONS

8.1 Acronyms

- LIMS:.....Laboratory Information Management System
- SOP:.....Standard Operating Procedure

8.2 Terminology

- Backup:.....To make a duplicate of a computer file or database on another device or tape to preserve existing work, in case of a computer failure or other mishap.
- error log:.....A file that receives error information whenever a program runs.
- execute:.....To carry out a program or a set of instructions.
- file:.....A collection of related information stored together on the system.
- full backup:.....A backup that includes all files on a device.
- incremental backup: A backup that includes only files changed or created since the last full or incremental backup. Files backed up incrementally reflect that they have been backed up.
- login:.....The process of identifying oneself to a computer.

9.0 ATTACHMENTS

Not applicable. (See SWL-GA-102 for record examples.)

SOUTHWEST LABORATORY OF OKLAHOMA, INC. *and*

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Standard Operating Procedure

Homogenization of Soil, Sediment, and Sludge Samples

Document No.: SWL-GA-140

Rev No. 1 Date: Rev 1.1 - 01/18/00

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APPROVALS	DATES
Chuck Hoover Procedure Prepared By	1/31/2000 Date
Steve E. Mathis Laboratory Operations Manager or Director	1/25/00 Date
Bryan Hicks Laboratory Safety Officer	1/25/00 Date
Chuck Hoover Laboratory QA/QC Officer	1/18/2000 Date

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

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HOMOGENIZATION OF SOILS, SEDIMENTS, and SLUDGES

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- 13.2 SWL-GA-114 2

14.0 DEFINITIONS – N/A 2

1.0 SCOPE and APPLICATION

1.1 The scope of this document is to detail the procedures used to obtain representative samples from soils, sediments, and sludges for all analyses except where volatile compounds are to be analyzed.

1.2 The procedures described herein will apply to all technicians involved in the preparation of sample media prior to analysis. Generally this will include the Sample Custodians, Extractions Chemists/Technicians, and/or Inorganic Chemists/Technicians

2.0 METHOD SUMMARY – N/A

3.0 HEALTH and SAFETY – N/A

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE – N/A

5.0 INTERFERENCE and POTENTIAL PROBLEMS

5.1 Care must be used when handling any sample since they may contain hazardous materials.

5.2 Take measures to ensure that equipment is scrupulously cleaned.

5.3 Work space areas shall be decontaminated between samples. It is recommended that liners be used as they may help in keeping work areas clean.

6.0 EQUIPMENT/APPARATUS

6.1 Stainless Steel Scoops

6.2 500ml, Pre-cleaned Glass Jar

6.3 SS mixing bowls

6.4 Labels

6.5 Protection gear; 1) gloves; 2) lab coat; 3) safety glasses

7.0 REAGENTS – N/A

8.0 PROCEDURE

8.1 Mixing in the Sample Container. It is imperative for any analysis to start with a representative sub-sample to insure reliable results. Reliable mixing can be achieved for the majority of soils and sediments by utilizing a scoopula and the sample container which the sample came in.

8.1.1 The analyst is to observe the consistency of the sample and mix throughout. Once mixed obtain a sub-sample from the entire sample.

8.1.2 Sub-samples (which are to be weighed) will be targeted to a "weight range" and not a predetermined specific weight (i.e.; 1 to 2 grams not 1.0 grams OR a range of 30 to 35 grams not 30.0 grams). By targeting a specific weight there could be a bias in the results due to particle size.

8.2 If the sample cannot be mixed in the sample container, the sample is to be transferred to a larger vessel, such as a quart SS mixing bowl.

8.2.1 Using a Scoop, breakup the sample into uniform sizes (approximately 4mm or less fragments) and homogenize in the bowl taking care to remove any artifacts such as vegetation or rocks.

8.2.2 After thoroughly mixing – obtain a representative portion of the sample for the preparation step.

8.3 Lab Duplicates or Matrix Spikes. When LCS/LCSD or MS/MSD's are to be analyzed, the analyst, after mixing, shall obtain both sub-samples from the same bowl.

9.0 CALCULATIONS – N/A

10.0 QUALITY ASSURANCE / QUALITY CONTROL – reference *SWL-GA-100*

11.0 WASTE DISPOSAL – reference *SWL-GA-114*

12.0 METHOD PERFORMANCE – N/A

13.0 REFERENCES

13.1 All QA/QC issues shall be referenced to SWL-GA-100, "*Laboratory Quality Assurance Program*".

13.2 Waste Disposal is governed by document SWL-GA-114, *"Hazardous Waste Management Plan"*

14.0 DEFINITIONS – N/A

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The Standard Operating Procedures (SOPs) contained herein are adopted by Pace Analytical Services.

These SOPs will be followed until superceded by Pace Analytical Services SOPs.

Signature:

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Quality Assurance Officer
Pace Analytical Services, Inc.
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Roger C. Splinter
Dir. of Quality, Safety, Training
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Dave Neal
General Manager
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Lenexa, KS

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Standard Operating Procedure

Anions in Water by Ion Chromatography

Document No.: SWL-IN-301
Rev No.: Date: Rev. 5.4 — 08/18/03

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APPROVALS	DATES
David L	8-18-03
Procedure Prepared By	Date
[signature]	8/19/3
Program Manager or Section Supervisor	Date
Chuck Horner	8/20/03
Laboratory Safety Officer	Date
Chuck Horner	8/20/03
Laboratory QA/QC Officer	Date

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

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ANIONS IN WATER BY ION CHROMATOGRAPHY

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Attachment 1 – Anions in Water Standard Batch Form 7

1.0 SCOPE AND APPLICATION

1.1 This method covers the determination of the following inorganic anions; Bromide, Chloride, Nitrate-N, Nitrite-N, Ortho-Phosphate-P, Sulfate, and Fluoride. This procedure covers EPA methods 300.0 and 9056. All QC requirements of each method are covered using the stricter requirement where applicable.

1.2 This method is applicable to the determination of the above anions in drinking water, surface water, mixed domestic and industrial wastewaters, groundwater, reagent waters, and solids (after extraction).

1.3 This method is recommended for use only by or under the supervision of analysts experienced in the use of ion chromatography and in the interpretation of the resulting chromatogram. Each analyst must demonstrate the ability to generate acceptable results with this method.

1.4 When this method is used to analyze unfamiliar samples for any of the above anions, anion identification should be supported by the use of sample matrix spikes covering the anions of interest.

2.0 METHOD SUMMARY

2.1 A 1.0 milliliter volume of sample is introduced into the ion chromatograph. The anions of interest are separated and measured, using a system comprised of a guard column, separator column, suppressor device, and conductivity detector.

2.2 In order to use this method for solids, an extraction procedure must be performed.

3.0 HEALTH AND SAFETY

See Standard Operating Procedure "*Laboratory Safety Plan*," SWL-GA-111.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING & STORAGE

4.1 Samples should be collected in scrupulously clean glass or polyethylene bottles.

4.2 Sample preservation and holding times for the anions that can be determined by this method are as follows:

Analyte	Preservation	Holding Time
Bromide	None required	28 days
Chloride	None required	28 days
Nitrate-N	Cool to 4°C	48 hours
Nitrite-N	Cool to 4°C	48 hours
O-Phosphate-P	Cool to 4°C	48 hours
Sulfate	Cool to 4°C	28 days
Fluoride	Cool to 4°C	28 days

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

5.1 Interferences can be caused by substances with retention times that are similar to and overlap those of the anion of interest. Large amounts of an anion can interfere with the peak resolution of an adjacent anion. Sample dilution and/or spiking can be used to solve most interference problems.

5.2 Method interferences may be caused by contaminants in the reagents water, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or elevated baseline in ion chromatograms.

5.3 Samples that contain particles larger than 0.45 microns and reagent solutions that contain particles larger than 0.20 microns require filtration to prevent damage to instrument columns and flow systems.

6.0 EQUIPMENT / APPARATUS

6.1 Balance – M ettler/H80, capable of accurately weighting to the nearest 0.0001g.

6.2 Ion Chromatograph – Dionex Model 2010i or DX300 with required accessories including syringes, analytical columns, compressed gases, and detectors.

6.3 Anion guard column – Dionex Ion Pac AG14-4mm.

6.4 Anion separator column – Dionex Ion Pac AS14-4mm

6.5 Anion suppressor device –Dionex Anion Micro-Membrane Suppressor (P/N 43074).

6.6 Detector – Dionex Conductivity Cell.

6.7 VG Chromatography server and mini-chrom data system.

6.8 The width of the retention time window used to make identifications is based on measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time is used to calculate a suggested window size for a compound.

7.0 REAGENTS

7.1 Reagent water – Deionized water, free of the anions of interest. The water must be filtered through a 0.20-micron filter.

7.2 Eluent Solution – Sodium bicarbonate 0.5 M, sodium carbonate 0.5 M. Dissolve 26.5g of sodium carbonate (Na_2CO_3) per 500 ml reagent DI water. Dissolve 21.25g sodium bicarbonate ($NaHCO_3$) per 500 ml reagent water. Use 14ml of the sodium carbonate 0.5 M solution and 4 ml of the sodium bicarbonate 0.5 M solution in a 2000ml volumetric flask and bring up to volume with reagent water.

7.3 Regeneration solution – Sulfuric acid, 0.2 N. Dilute 5.6ml conc. sulfuric acid (H_2SO_4) to 1 liter with reagent water.

7.4 Stock Standard solutions, 1000 mg/l – Certified stock standards for all anions are purchased from NSI Industries, Inc. (or another NIST-certified manufacturer), and are the bases for all working standards.

7.5 Intermediate Standard – Dilute the 1000ppm stock standards to 500ml according to the following schedule: 25ml Br^- (50ppm), 50ml Cl^- (100ppm), 25mls NO_3^- (20ppm), 10ml NO_2^- (20ppm), 10mls PO_4^- (20ppm), 10ml F^- (20ppm), and 50ml SO_4^- (100ppm).

7.6 Working Standards (Method 2010i):

7.6.1 Number 2 Standard – Dilute 125ml of the Intermediate Standard (Section 7.5) to 250ml in reagent water to obtain the following concentrations: 25ppm Br^-, 50ppm Cl^-, 10ppm NO_3^-, 10ppm NO_2^-, 10ppm PO_4^-, 10ppm F^-, and 50ppm SO_4^-.

7.6.2 Number 3 Standard – Dilute 25ml of the Intermediate Standard (Section 7.5) to 250ml in reagent water to obtain the following concentrations: 5ppm Br^-, 10ppm Cl^-, 2ppm NO_3^-, 2ppm NO_2^-, 2ppm PO_4^-, 2ppm F^-, and 10ppm SO_4^-.

7.6.3 Number 4 Standard – Dilute 1ml of the Intermediate Standard (Section 7.5) to 100ml in reagent water to obtain the following concentrations: 0.5ppm Br^-, 1.0ppm Cl^-, 0.2ppm NO_3^-, 0.2ppm NO_2^-, 0.2ppm PO_4^-, 0.2ppm F^-, and 1.0ppm SO_4^-.

7.6.4 Number 5 Standard – Dilute 0.5ml of the Intermediate Standard (Section 7.5) to 100ml in reagent water to obtain the following concentrations: 0.25ppm Br^-, 0.5ppm Cl^-, 0.1ppm NO_3^-, 0.10ppm NO_2^-, 0.10ppm PO_4^-, 0.10ppm F^-, and 0.5ppm SO_4^-.

7.7 Initial Calibration Verification – A certified premixed standard is purchased from SCP Science (or another NIST-certified manufacturer) and should have the following approximate concentrations: 25ppm Br^-, 50ppm Cl^-, 10ppm NO_3^-, 10ppm NO_2^-, 10ppm PO_4^-, 10ppm F^-, and 50ppm SO_4^-.

7.8 Stability of standards. Stock standards are stable for at least 1 month when stored at 4° C. Diluted working standards should be prepared weekly, except those that contain nitrite and phosphate, which should be prepared fresh daily.

8.0 PROCEDURE

8.1 Instrument analysis – Refer to the procedure as listed in the instrument manual.

8.2 Calibration and Standardization

8.2.1 Inject 1.0ml of the mid-range calibration standard.

8.2.2 Using the retention times from this standard, build your calibration table entering the concentrations for each standard, in Calibration Menu of Dionex Mini-chrome software.

8.2.3 Using injection of 1.0ml of each calibration standard, tabulate peak area against the concentration. The results will be used to prepare a calibration curve for each analyte. The coefficient for each anion should be 0.995 or better in the Cal Plot Menu of the mini-chrome software. Calibration curve should be in linear and no force zero.

8.2.4 A minimum of three calibration standards must be used. A calibration is required at a minimum of once a month.

8.2.5 The calibration curve must be verified on each working day, or whenever the anion eluent is changed, and after every 10 samples. If the response or retention time for any analyte varies from the expected values by more than ±10%, the test must be repeated, using fresh calibration standards. If the results are still more than ±10%, a new calibration curve must be prepared for that anlayte.

8.3 Analysis

8.3.1 Start the system and prime the column by injecting a standard. Then check the calibration as described in Section 8.2.

8.3.2 Load and inject 1.0 – 2.0ml of well mixed samples. Flush the injection loop thoroughly, using each new sample. Use the same size loop for standards and samples.

8.3.3 The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards.

8.3.4 If the response for the peak exceeds the working range of the system, dilute the sample with an appropriate amount of reagent water and reanalyze.

8.3.5 If the resulting chromatogram fails to produce adequate resolution, or if identification of specific anions is questionable, fortify the sample with an appropriate amount of standard and reanalyze.

8.3.6 The following extraction should be use for solid materials. Weigh 10.0g of sample into a plastic 4oz sample container. Add 100ml of deionized water. Shake for 10-20 minutes in a mechanical shaker. Pour slurry into a 40ml VOA vial, and centrifuge for 30min. Filter the supernatant through a 0.2 micron I.C. filter, and load autosampler as normal.

8.4 Write-up and review

8.4.1 Transfer electronic data to the inorganic database, print out run log, complete write-up, and upload data to LIMS. Refer to Inorganic Database SOP SWL-IN-108 for procedure and forms.

8.4.2 Review data according to the guidelines in Section 10.0 and 12.0 of this SOP and "1st and 2nd Data Review for Wet Chemistry" SOP SWL-IN-107.

9.0 CALCULATIONS

9.1 Results are printed in mg/l. No calculations are necessary for waters.

9.2 For Soils:

The instrument reading is in mg/l, the conversion is as follows:

$$mg/kg = \frac{(\text{Instrument reading in mg/l}) * (\text{Final volume in Liters})}{(\text{Initial weight in grams}) * (1kg/1000g)}$$

10.0 QUALITY ASSURANCE / QUALITY CONTROL

10.1 Immediately after the calibration has been established, the calibration must be verified for every analyte by the analysis of an Independent Standard (ICV). When measurements exceed the 90-110% confidence interval, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration reverified.

10.2 A Calibration Verification (CCV) and a Calibration Blank (CCB) must be ran every 10 analytical samples and after the last sample. The CCV must agree within 90-110% recovery of the expected value with less than a 5% variation from the previous or subsequent CCV, and the CCB must be below the reporting limit. If they are not, terminate the analysis, correct the problem, and recalibrate the instrument.

10.3 The following QC samples must be analyzed with every batch of 20 samples per matrix

10.3.1 Preparation Blank – result must be below the Reporting limit for the analyte of interest, or the analysis must be terminated and samples re-prepared.

10.3.2 Laboratory Control Sample and Laboratory Control Sample Duplicate – Measurements must be within 90-110% recovery, or the analysis must be terminated and samples re-prepared.

10.3.3 Matrix Spike and Matrix Spike Duplicate – If Measurements fall outside of 80-120% R the data will be flagged. The following procedure will be followed for matrix spikes:

10.3.3.1 Waters – Add 50µl of a 1,000mg/l stock standard for each analyte and bring up to a final volume of 5ml with the sample, giving a spike value of 10 mg/l.

10.3.3.2 Soils – Add 1.0ml of 1,000 mg/l stock standard for each analyte to 10g of sample. Follow the soil extraction procedure in Section 8.3.6, thus giving a 10 mg/l or 100 mg/kg spike value.

10.4 Reprocessing of QC integrations should be avoided. When performed, they must be printed out, signed by the analyst attached to a Non-Conformance Report and reviewed by the supervisor. The Non-Conformance is to be discussed in the case Narrative and a copy of the report is to be included with the data to the client.

11.0 WASTE DISPOSAL/POLLUTION PREVENTION

Refer to SOP SWL-GA-114, "*Hazardous Waste Management Plan.*"

12.0 METHOD PERFORMANCE

12.1 Preparation Blank – result must be below the Reporting limit for the analyte of interest, or the analysis must be terminated and samples re-prepared.

12.2 Laboratory Control Sample and Laboratory Control Sample Duplicate – Measurements must be within 90-110% recovery, or the analysis must be terminated and samples re-prepared.

12.3 Matrix Spike and Matrix Spike Duplicate – If Measurements fall outside of 80-120% R the data will be flagged. The following procedure will be followed for matrix spikes:

12.3.1 Waters – Add 50µl of a 1,000mg/l stock standard for each analyte and bring up to a final volume of 5ml with the sample, giving a spike value of 10 mg/l.

12.3.2 Soils – Add 1.0ml of 1,000 mg/l stock standard for each analyte to 10g of sample. Follow the soil extraction procedure in Section 8.3.6, thus giving a 10 mg/l or 100 mg/kg spike value.

13.0 REFERENCES

13.1 U.S. Environmental Protection Agency, Methods for the Determination of Inorganic Anions By Ion Chomatography, Method 300.0, Rev. 2.1, August 1993.

13.2 Standard Methods for the Examination of Water and Wastewater, 18th Edition, APHA-AWWA-WPCF, Method 4110, pp. 4-1 to 4-5.

13.3 SW846 Method 9056 Rev. 0, September 1994.

13.4 Southwest Laboratory Standard Operating Procedures listed in text.

14.0 DEFINITIONS

None required. Terminology is explained in text.

15.0 ATTACHMENTS

Attachment 1: Anions in Water Standard Batch Form

ATTACHMENT 1
Anions in Water Standard Batch Form

ANIONS IN WATER STANDARD BATCH FORM

ANIONS STD. BATCH FORM (IN301F1.DOC) SWL-IN-301, REV. 5.1 11/26/01

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

REAGENTS FOR ANIONS IN WATER

REAGENT	SOLUTION ID #
Eluent Solution	_____
Sulfuric Acid	_____

SOLUTION ID

Solution	Solution ID #
ICV	_____
Matrix Spike	_____
Std. #1/ Internal Std.	_____
Std. #2/ CCV and LCW, LDW	_____
Std. #3	_____
Std. #4	_____
Std. #5/ CRA	_____

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Standard Operating Procedure

Nitrate-Nitrite/Nitrite (Colorimetric, Automated, Cadmium Reduction)

Document No.: SWL-IN-304

Rev No. / Date: Rev. 6.0 — 07/11/02

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APPROVALS	DATES
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Laboratory QA/QC Officer	Date

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NITRATE-NITRITE/NITRITE (COLORIMETRIC, AUTOMATED, CADMIUM REDUCTION)

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1.0 SCOPE AND APPLICATION

1.1 This method pertains to the determination of nitrite singly, or nitrite and nitrate combined in surface and saline waters, and domestic and industrial wastes.

1.2 The applicable range of nitrate-nitrite nitrogen for this method is 0.1 to 2.0mg/l for waters or 1.0 to 20.0mg/kg for soils. The range may be extended with sample dilution.

1.3 This SOP (Standard Operating Procedure) covers USEPA Method 353.2.

1.4 This procedure contains no method deviations.

2.0 METHOD SUMMARY

A filtered sample is passed through a column containing granulated copper-cadmium to reduce nitrate to nitrite. The nitrite that was originally present plus reduced nitrate is determined by diazotizing with sulfanilamide and coupling with n-(1-naphthyl)-ethylenediamine dihydrochloride to form a highly colored azo dye which is measured colorimetrically. Separate, rather than combined nitrate-nitrite, values are readily obtained by carrying out the procedure first with, and then without the cu-cd reduction step.

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. This may be done by wearing gloves, eye protection and laboratory coat during analysis.

3.3 The following chemicals have the potential to be highly toxic or hazardous, consult MSDS: phosphoric acid, n-1-naphthylethylene diamine dihydrochloride, hydrochloric acid and copper sulfate.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING & STORAGE

4.1 Samples should be collected in plastic or glass bottles. All bottles must be thoroughly cleaned and rinsed with reagent water.

4.2 Samples for nitrate/nitrite should be preserved with sulfuric acid (2ml conc. H_2SO_4 per liter) and refrigerated at 4°C. Samples must be analyzed within 28 days after sample collection.

4.3 Samples for nitrite alone must be stored at 4°C and analyzed within 48 hours.

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

5.1 Build up of suspended matter in the reduction column will restrict sample flow. Since nitrate-nitrogen is found in a soluble state, the sample may be pre-filtered.

5.2 Low results might be obtained for samples that contain high concentrations of iron, copper, or other metals. EDTA is added to the samples to eliminate this interference.

5.3 Samples that contain large concentrations of oil and grease will coat the surface of the cadmium. Pre-extracting the sample with an organic solvent eliminates this interference.

6.0 EQUIPMENT/APPARATUS

6.1 Lachet QuikChem Automated Flow Injection Ion Analyzer which includes:

6.1.1 Automatic Sampler (with glass sample tubes)

6.1.2 Proportioning Pump

6.1.3 Injection Module with a 17cm x 0.8mm sample loop

6.1.4 Colorimeter with:

6.1.4.1 Interference Filter: 520nm

6.1.4.2 Flow cell: 10mm, 80uL

6.1.5 Reaction Module 10-107-04-1-C with cd column

6.1.6 QuikCalc II Software System

6.2 100ml volumetric flask

6.3 Class A volumetric pipettes

6.4 100ml plastic snap-top container.

7.0 REAGENTS

7.1 *Cadmium granules*: 0.3 – 1.5mm diameter, Lachet Part No. 5000-231.

7.2 *Copper-cadmium*: The cadmium granules are cleaned with dilute HCl and copperized with 2% solution of copper sulfate in the following manner:

7.2.1 Wash the cadmium with HCl and rinse with distilled water. The color of the cadmium so treated should be silver.

7.2.2 Swirl 10g cadmium in 100mL portions of 2% solution of copper sulfate for five minutes or until blue color fades, decant and repeat with fresh copper sulfate until brown colloidal precipitate forms.

7.2.3 Wash the cadmium-copper with distilled water (at least 10 times) to remove all the precipitated copper. The color of the cadmium so treated should be black.

7.2.4 At least one nitrite standard should be compared to a reduced nitrate standard at the same concentration to verify the efficiency of the reduction column. The Cu-Cd granules should be reactivated when recovery falls below 75%.

7.3 *Preparation of reduction column:*

7.3.1 Clamp the empty column upright so that your hands are free.

7.3.2 Unscrew one of the colored fittings from an end of the column, and pull out and save the foam plug.

7.3.3 Fasten this fitting up higher than the open end of the column and completely fill the column, attached fittings, and tubing with ammonium chloride buffer.

7.3.4 Scoop up copperized cadmium granules prepared in section 7.2 with a spatula, and pour them into the top of the water-filled column so that they float down to the bottom of the column. Continue pouring the cadmium in and tapping the column with a screwdriver handle to dislodge any air bubbles and to prevent gaps in the cadmium filling. When the cadmium granules reach to about 5mm from the open end of the column, push in the foam plug and screw on the top fitting.

7.3.5 Rinse the outside of the column with water. If air remains in the column or is introduced accidentally, connect the column into the manifold, turn the pump on maximum, and tap firmly with a screwdriver handle, walking up the column until all air is removed.

7.4 *Color reagent*: To approximately 800 ml of Distilled water add, while stirring, 100ml conc. phosphoric acid, 40g sulfanilamide, and 1g N-1-naphthylethylene diamine ____ dihydrochloride. Stir until dissolved and dilute to 1 liter. Store in a brown bottle and keep in the dark when not in use. This solution is stable for several months.

7.5 *Dilute hydrochloric acid, 6N*: Dilute 50ml of conc. HCl to 100ml with distilled water.

7.6 *Copper sulfate solution 2%*: Dissolve 20g of $CuSO_4 \bullet 5H_2O$ in 500ml of distilled water and dilute to 1 liter.

7.7 *Ammonium chloride buffer*: In a hood, to a 1-liter volumetric flask add 500ml distilled water, 105ml conc. HCl, 95ml ammonium hydroxide (NH_4OH) and 1.0g disodium EDTA. Dilute to 1-liter.

7.8 *Stock nitrate solution*: NSI Ion Standard, 10,000ug/mL. Certificate of Analysis on request.

Note: Keep stock standard refrigerated and do not use after the manufacturers recommended expiration date.

7.9 *Stock nitrite solution*: NSI Ion Standard, 10,000 ug/mL. Certificate of Analysis on request.

Note: Keep stock standard refrigerated and do not use after the manufacturers recommended expiration date.

7.10 *Standard nitrate solution*: Dilute 1.0ml of stock nitrate (see section 7.8) to 100ml.

7.11 *Standard nitrite solution*: Dilute 1.0ml of stock nitrite (see section 7.9) to 100ml.

7.12 *Using standard nitrate solution* (see section 7.10) *prepare the following standards in 100.0mL volumetric flasks.* At least one nitrite standard should be compared to a nitrate standard at the same concentration to verify the efficiency of the reduction column. Write all standard preparations in the **Standard Preparation Log, form 104.ATT3.doc**, found in SOP "*Inorganic Traceability of Standards*", SWL-IN-104.

Conc. mg NO_2-N or NO_3-N/1	ml Standard Solution/100mL
0.0	0.0
0.1	0.1
0.5	0.5
1.0	1.0
2.0	2.0

7.13 2 M KC1 Solution: 150 KCL/L

8.0 PROCEDURE

8.1 Injection Timing

8.1.1 Pump Speed: 35

8.1.2 Cycle Period: 40s

8.1.3 Load Period: 20s

8.1.4 Inject Period: 20s

8.2 Nitrate + Nitrite

8.2.1 Inject to start of peak period: 22s

8.2.2 Inject to end of peak period: 58s

8.3 Nitrite (no column)

8.3.1 Inject to start of peak period: 15s

8.3.2 Inject to end of peak period is 51s

8.3.3 Gain = 260 x 1

8.4 System Operation

8.4.1 Inspect modules for proper connections. (See Figure 1).

8.4.2 Turn on power and all modules, except sampler.

8.4.3 Place reagent feed-lines into proper containers. Raise tension levers on pump tube cassettes.

8.4.4 Turn on sampler and allow reagents to pump through the system.

8.4.5 Stop the pump and place reduction column in the manifold. Start the pump.

8.4.6 Pump system until a stable baseline is attained.

8.4.7 Place calibration standards and blank in sample tray in descending order of concentration followed by unknowns and check standards. Submit tray and start run.

8.4.8 At end of run, turn off the pump and remove reduction column from manifold and connect ends with a union, taking care to avoid introduction of air bubbles.

8.4.9 Turn on the pump and place all feed-lines in water. Flush system and pump dry.

8.4.10 Turn off pump, all modules and release levers on pump tube cassettes.

SYSTEM NOTE: The CD column efficiency should be above 90% if working properly. To check this, inject one or several different nitrite standards followed by one or several different nitrate standards. Fit the nitrite standards response to a line. The ratio of the slope of the nitrate/nitrite lines x 100% is the efficiency of the column.

8.4.11 Transfer electronic data to the Inorganic Database, print run log, complete write-up and upload data to LIMS. Refer to "*Inorganic Database*", SWL-IN-108 for procedure and forms. If data is not available in electronic form, print run log from instrument and submit for review.

8.5 Soil Preparation

8.5.1 Weigh 10g of soil and place in a snap-cap container.

8.5.2 Add 100ml of 2M KCl.

8.5.3 Cap flask and shake for one (1) hour.

8.5.4 Filter through a Whatman 4 filter.

8.6 Nitrite may be measured by the above procedure by omitting the cadmium reduction column used in step 8.4.5.

9.0 CALCULATIONS

9.1 Calculate the nitrate-nitrite, in mg/kg as follows:

$$\text{mg/kg } NO_3\text{-}NO_2 \text{ (wet weight)} = \frac{(A \times 0.01)}{B} \times 1000$$

Where: A = mg/l NO_3-NO_2 from analysis
B = wet weight of original sample in grams.

9.2 Water samples are calculated by the instrument, in mg/l.

9.3 Nitrate-Nitrite results are calculated directly from the instrument using the cadmium reduction column.

9.4 Nitrite results are calculated directly from the instrument by analyzing samples without the cadmium reduction column.

9.5 Nitrate can be determined by subtracting the results obtained by using the cadmium reduction column, from the results obtained by not using the column.

10.0 QUALITY ASSURANCE/QUALITY CONTROL

10.1 Sample Batch QA/QC Requirements.

10.1.1 Prepare method blank per sample batch and take through the entire analytical process. If NO_3-NO_2 is detected, the entire batch will need to be reanalyzed. A sample batch can consist of up to twenty samples per matrix.

10.1.2 Prepare a Laboratory Control Spike (LCS) and an LCS Duplicate (LCSD) per sample batch, to determine the precision and accuracy of the method. The percent recovery of the LCS should be within 90-110%. If recovery is outside this range on both the LCS and the LCSD, then the analytical process is out of control and the entire batch must be reanalyzed. The precision Relative Percent Difference (RPD) between the LCS and the LCSD should be less than twenty percent.

$$RPD = \frac{|LCS - LCSD|}{(LCS + LCSD)/2} \times 100$$

10.1.3 One set Matrix Spike (MS) and Matrix Spike Duplicate (MSD) analysis will be performed per 20 samples or batch. Percent Recovery must be within 90-110%, and RPD must be <20. All data outside of these ranges must be flagged to indicate possible matrix interference.

10.1.4 LCS/LCSD/MS/MSD spiking instructions:

50uL of 100ppm NO_3-standard and 50uL of 100ppm NO_2 standard to 10ml of DI water (for LCS/LCSD) or 10ml of sample (for MS/MSD).

10.2 Initial Demonstration of Performance

10.2.1 MDL Study: Follow the procedure as stated in SOP SWL-GA-113, "*Definition and Determination of Detection Limits*". This will be done on a yearly basis. The PQL (or reporting limit) will be based on 2-3x the MDL.

10.3 Calibration Checks

10.3.1 Initial Calibration Verification (ICV): After the system has been calibrated (with a Linear Correlation Coefficient of >0.995), the accuracy of the calibration shall be verified with a second source standard, which is from a different manufacturer or lot number than the calibration standards.

10.3.2 Calibration Blank (ICB or CCB) – A calibration blank is analyzed after each ICV and CCV. Blanks are to be reported down to the MDL.

10.3.3 Continuing Calibration Verification (CCV) – To assure calibration accuracy during each analysis run, the mid-level calibration standard will be analyzed at a frequency of 10% and after the last analytical sample.

10.3.4 CRA – To assure calibration at the low end of the calibration curve. This low standard will be analyzed at the beginning of each run.

10.4 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 the "*Hazardous Waste Management Plan*," SOP SWL-GA-114.

12.0 METHOD PERFORMANCE

12.1 ICV/CCV – Must have a 90-110% recovery of the true value. If not, terminate the analysis, correct the problem, re-calibrate and re-analyze all associated samples.

12.2 ICB/CCB – Must be below the PQL (0.1mg/l). If not, terminate the analysis, correct the problem, re-calibrate and re-analyze all associated samples.

12.3 CRA – Should have a 70-130% recovery of the true value. If not, all samples near the low end of the calibration curve should be considered suspect and re-analyzed.

12.4 PBW/PBS – Must be below the PQL (0.1mg/l). If not, re-prepare and analyze all samples that are not >10x the PBW/PBS value.

12.5 LCW/LDW/LCS/LDS – Must have a 90-110% recovery of the true value. If not, re-prepare and analyze all samples associated in the batch.

12.6 Matrix Spike and Matrix Spike Duplicate (MS/MSD) – Should have a 90-110% recovery. If not, flag all associated data with a "N".

13.0 REFERENCES

13.1 U.S. EPA, "Methods for Chemical Analysis of Water and Wastes", Method 353.2, Storet No. Total 00630.

13.2 "Methods for Determination of Inorganic Substances in Water and Fluvial Sediment", Book 5, Chapter A1, U.S. Department of the Interior, U.S. Geological Survey, Revision 04/11/89.

13.3 Lachat Instruments Operating Manual

14.0 DEFINITIONS

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.

CRA Standard:.... A standard to assure the calibration of the instrument is working at the low end of the calibration curve.

ICV:..... Initial Calibration Verification: A standard that is from a different source than the calibration standards, and used to verify the calibration.

ICB:..... Initial Calibration Blank: Instrument blank, taken through analysis to check for contamination in the system.

CCB: ..... Continuing Calibration Blank: Instrument blank, taken through analysis to check for contamination in the system.

CCV: ..... Continuing Calibration Verification: Mid-range standard used to verify the analytical sequence is still within control.

Red Book: ..... SWL book, which consists of all the MDL/PQL per method for the laboratory.

MDL:..... Method Detection Limit: A detection limit derived from seven samples taken through the entire method.

PQL:..... Practical Detection Limit: SWL reporting limit based on a factor times the MDL.

Sample Batch: ..... Consists of a maximum of twenty samples per matrix.

Preparation Blank (water-PBW, soil-LCS): Method blank, taken through preparation and analysis to check for contamination in the system.

Laboratory Control Sample (water-LCW, soil-LCS): A spiked sample (usually water) taken through preparation and analysis to check the system's accuracy.

Laboratory Duplicate Control Sample (water-LDW, soil-LDS): A spiked sample (usually water) taken through preparation and analysis to check the system's accuracy and precision.

MS:..... Matrix Spike: A spiked sample taken through preparation and analysis to check the accuracy of the matrix.

MSD:..... Matrix Spike Duplicate: A spiked sample taken through preparation and analysis to check the accuracy and precision of the matrix.

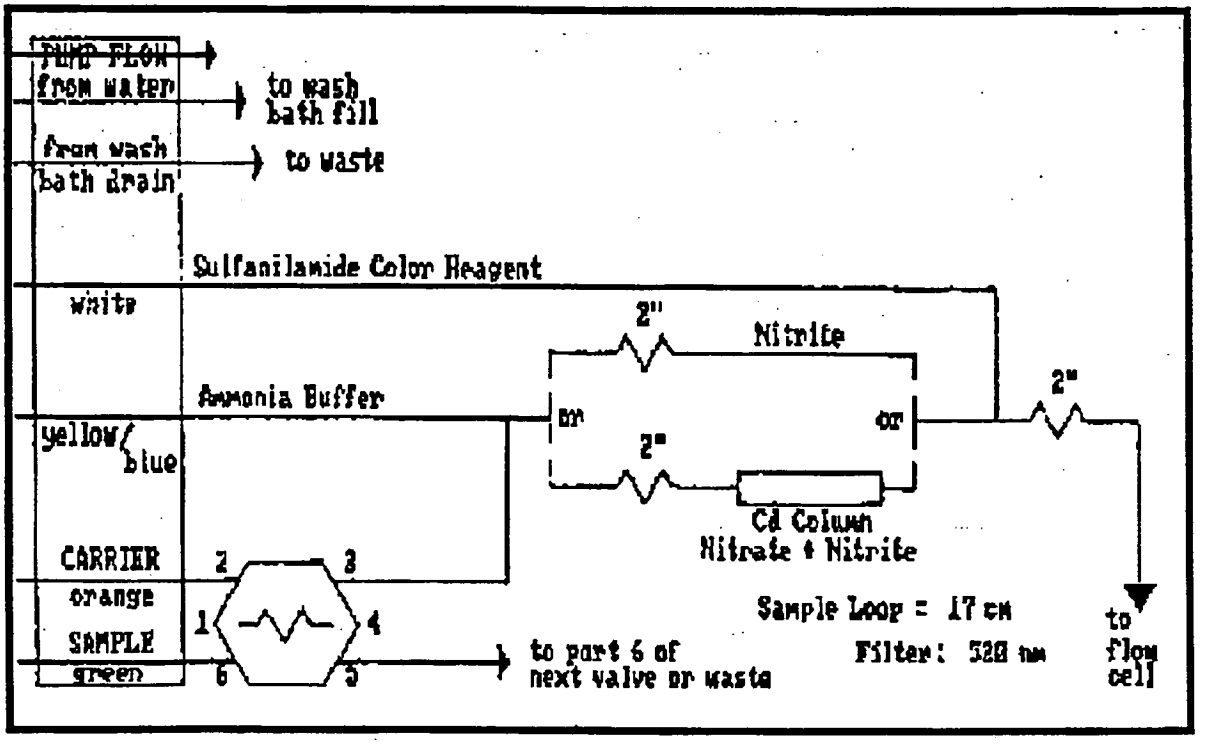
Duplicate Sample: A sample taken through preparation and analysis to check the precision of the matrix.

15.0 ATTACHMENTS

Figure 1: Manifold Diagram

Attachment 1: Nitrate-Nitrite/Nitrite Standard Batch Form

FIGURE 1
Manifold Diagram



CARRIER is water. 1" is 70.0 cm of tubing on a 1 inch coil support 2" is 135 cm of tubing on a 2 inch coil support 2.5" is 168 cm of tubing on a 2.5 inch coil support 3" is 202 cm of tubing on a 3 inch coil support 4" is 255 cm of tubing on a 4 inch coil support 8" is 550 cm of tubing on a 8 inch coil support	Heating tubing is shown inside a box with the temperature next to the box. Heated tubing is 650 cm unless otherwise specified. All manifold tubing is 0.8 mm (0.032 in) i.d. This is 5.2 μL/cm. NOTES: A new manifold includes two packed cadmium columns. MANIFOLD DIAGRAM REVISION DATE: 30 Dec 1986

ATTACHMENT 1
Nitrate-Nitrite/Nitrite Standard Batch Form

Analyst: _____ Date: _____ Standards Batch ID: _____

Reagents for Nitrate-Nitrite/Nitrite

Reagent	Lot # / Unique Id #
Ammonium Chloride Buffer	_____
Sulfanilamide Color Reagent	_____

STANDARD PREPARATION

Intermediate:

NO_3/NO_2: _____ Date Expired: _____

NO_2: _____ Date Expired: _____

Calibration Standards

mL 100mg/L Intermediate stock solution per 100mL	Final Conc.
0.1	0.10
0.5	0.50
1.0	1.00
2.0	2.00

Laboratory Control Sample; (0.5mg/L): 50uL from Intermediate stock solution per 10mL sample.
Matrix Spike; (1.0mg/L): 50uL from Intermediate stock solution per 10mL sample.

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The Standard Operating Procedures (SOPs) contained herein are adopted by Pace Analytical Services.

These SOPs will be followed until superceded by Pace Analytical Services SOPs.

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

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Standard Operating Procedure

Analysis of Total Organic Carbon in Water

Document No.: SWL-IN-310

Rev No. / Date: Rev. 5.3 — 05/28/03

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APPROVALS	DATES
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Procedure Prepared By	Date
	5/28/3
Program Manager or Section Supervisor	Date
Chuck Haney	5/28/03
Laboratory Safety Officer	Date
Chuck Haney	5/28/03
Laboratory QA/QC Officer	Date

(Effective Date is 10 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 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
2.0	10.0
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. Air carrier flow rate should be approximately 140-150°.

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 Initial Calibration Standard (ICV), Continuing Calibration Standard (CCV), Initial 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
10.0	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 4; *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 quadruplicate 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 prepared 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-prepared.

10.4.2 Laboratory Control Samples (LCW and LDW) will be prepared with every batch. With a true value of 50.0 mg/l the LCW and LDW have a control limit of 90-110% recovery. If out of control, the samples will be re-prepared.

10.4.3 Initial 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 Initial 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 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-prepared for analysis.

12.3 Laboratory Control Samples (LCW and LDW) will be prepared with every batch. With a true value of 50.0 mg/l the LCW and LDW have control limit of 90-110% recovery. If out of control the samples will be re-prepared and analyzed.

12.4 Initial 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 Initial 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 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

MSDA:..... 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.

MDL:..... Method Detection Limit: A detection limit derived from seven samples taken through the entire method.

PQL:..... Practical Detection Limit: SWL reporting limit based on a factor times the MDL.

Sample Batch: ..... Consists of a maximum of twenty samples per matrix.

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Laboratory Control Sample (water-LCW): A spiked sample (usually water) taken through preparation and analysis to check the system's accuracy.

Laboratory Duplicate Control Sample (water-LDW): A spiked sample (usually water) taken through preparation and analysis to check the system's accuracy and precision.

MS:..... Matrix Spike: A spiked sample taken through preparation and analysis to check the accuracy of the matrix. This is designed to provide information about the effect of the sample matrix on the preparation and/or measurement methodology.

MSD:..... Matrix Spike Duplicate: A spiked sample taken through preparation 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 preparation and/or measurement methodology.

Duplicate Sample: A sample taken through preparation and analysis to check the precision of the matrix.

Redbook:..... A listing of all IDL, MDL, and PQL for the laboratory.

Bluebook:..... Summary listing of in-house control limits.

ICV:..... Initial Calibration of Verification Standard: 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.

CCV:..... Continuing Calibration Verification solution: A mid-range standard used to verify calibration throughout analysis.

ICB/CCB:..... Calibration Blanks: A standard blank used to verify calibration throughout analysis.

15.0 ATTACHMENTS

15.1 Figures

Figure 1: Front View of the TOC-505 Interior with Front Door Open

15.2 Attachments

Attachment 1: TOC Run Log

FIGURE 1
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.

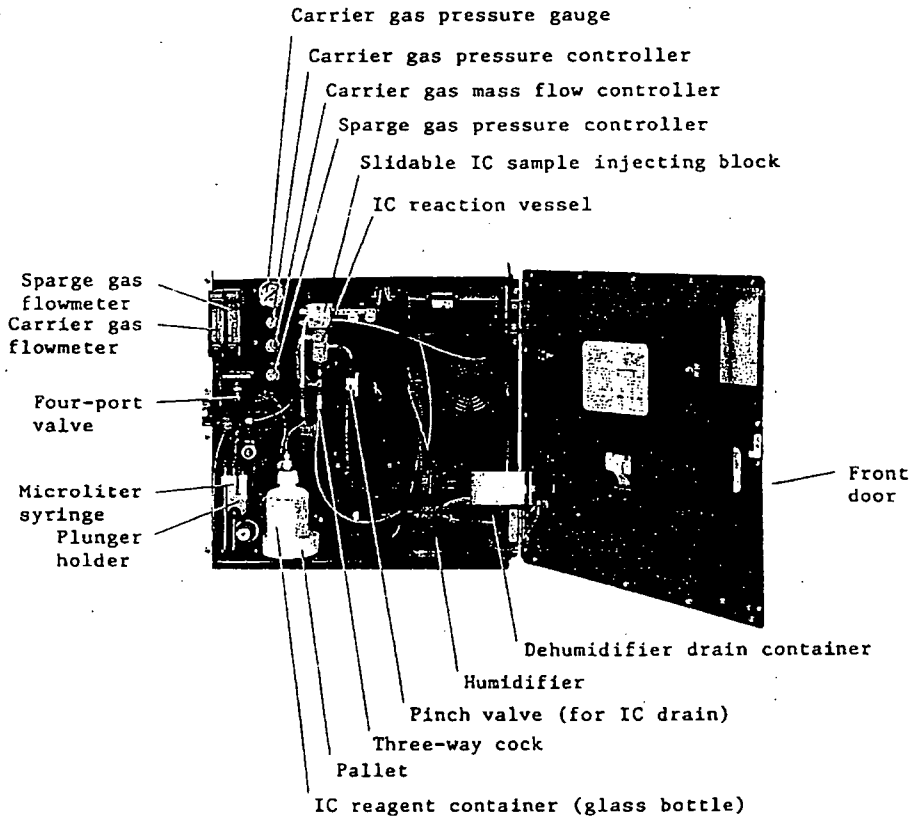

Fig. 4.1 Front View of the TOC-5050 Interior (Viewed with the Front Door Open)

ATTACHMENT 1
TOC Run Log

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

TM

TOC Run Log

INORGANIC DEPARTMENT

Book ID: IN310-TOC-___
Log Book START to FINISH DATE:

Ref: SWL-IN-310
In310a1cov

TOC Run Log, Ref: SWL-IN-310

TOC Run Log (IN310A1.DOC) SOP SWL-IN-310 Rev. #: 5.0 — 10/17/01 IN045, IN047, & IN048

Instrument: _____ Date of Last Calibration: _____ Batch ID #: _____

Analyst: _____ Date of Analysis: _____ Book #: IN310-TOC-_____

Instrument Sample #	SWLO Sample #	Time of Analysis	Dilution Factor	TC or TOC mg/L	TIC	Comments

Reviewed By: _____ Date: _____

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SOUTHWEST LABORATORY OF OKLAHOMA, INC. *and*
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TM

Standard Operating Procedure

Total Organic Carbon in Soil/Sediment

Document No.: SWL-IN-311

Rev No. / Date: Rev. 4.3 — 12/03/01

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APPROVALS	DATES
Kenneth A. Lynch Procedure Prepared By	12-3-01 Date
Program Manager or Section Supervisor	12-4-01 Date
Chuck Jones Laboratory Safety Officer	12/04/01 Date
Chuck Jones Laboratory QA/QC Officer	12/04/01 Date

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

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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, modified for soils.

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 See SOP SWL-GA-111, "*Laboratory Safety Plan.*"

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 Leco Calibration Standard: 1.00% carbon.

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.

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. Results should be within 2% of average value.

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. Use a minimum of three results. 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. Re-calibrate if calibration states it is out of range.

8.2.17 Analyze the LCS and LDS to verify the calibration. The percent recovery must be within ±15% or re-calibration will be necessary. 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 0.05% or re-calibration 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 in Soil/Sediment Log*" (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 Leco Standard, 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

See 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

ICV..... Initial Calibration Verification: A standard that is from a different source than the calibration standards, and is used to verify the calibration.

CCB: ..... Continuing Calibration Blank: Empty combustion boat taken through the analysis to check for contamination in the system.

CCV: ..... Continuing Calibration Verification: Standard used to verify the analytical calibration is still within control.

Sample Batch: Consists of a maximum of twenty samples per matrix.

PBS: ..... Preparation Blank: Method blank taken through sample preparation steps and analysis to check for contamination in the system.

LCS: ..... Laboratory Control Sample: A privately prepared sample of known concentration taken through the sample preparation and analysis to check the system's accuracy.

MS:..... Matrix Spike: A spiked sample taken through analysis to check the accuracy of the matrix.

MSD:..... Matrix Spike Duplicate: A spiked sample taken through analysis to check the accuracy and precision of the matrix.

15.0 ATTACHMENTS

15.1 Attachment 1: Total Organic Carbon in Soil/Sediment Log

ATTACHMENT 1
Total Organic Carbon in Soil/Sediment Log

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

TM

Total Organic Carbon in Soil/Sediment Log

INORGANIC DEPARTMENT

Book ID: IN311-TOC-___
Log Book START to FINISH DATE:

Ref: SWL-IN-311
In311L1cov

Total Organic Carbon in Soil/Sediment Log

DEPARTMENT

(IN311L1.DOC) SWL-IN-311, REV. 4.2 10/16/01 Book ID: IN311-TOC-_____

Batch ID: _____ **Test Code:** _____ **Date:** _____ **Time:** _____ **Weight:** _____

Analyst: _____ **Analysis Date:** _____

#	Client ID	SWLO NO	Weight of Sample, gm	TOC, mg/Kg Wet Weight	Initial Weight	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						

To Convert: % x 10,000 = mg/Kg

Reviewed By: _____ **Date:** _____

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The Standard Operating Procedures (SOPs) contained herein are adopted by Pace Analytical Services.

These SOPs will be followed until superceded by Pace Analytical Services SOPs.

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

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Standard Operating Procedure

Total Hardness: Titrimetric (IN140) and Calculation (IN145)

Document No.: SWL-IN-502

Rev No. / Date: Rev. 4.3 — 01/29/03

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APPROVALS	DATES
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TOTAL HARDNESS: TITRIMETRIC (IN140) AND CALCULATION (IN145)

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1.0 SCOPE AND APPLICATION

1.1 Hardness by the EDTA titration method is applicable to drinking, surface and saline water, domestic and industrial wastes. The method is suitable for all concentration ranges of hardness; however, in order to avoid large titration volumes, use a sample aliquot containing not more than 25mg $CaCo_3$.

1.2 Hardness by calculation is applicable to all waters and yields the higher accuracy. If a mineral analysis is performed, hardness by calculation can be reported.

1.3 Any deviations from the methodologies listed in Section 13.0 encompass the most stringent of the methods' QC requirements (see Sections 10.0 & 12.0).

1.4 The SOP covers USEPA method 130.2 and Standard Methods SM2340B and 2340C. All QC requirements of each are covered using the stricter requirement where applicable.

2.0 METHOD SUMMARY

2.1 Hardness by EDTA titration: Calcium and magnesium ions in the sample are segregated upon the addition of disodium ethylene diamine tetracetate (EDTA). The end point of the reaction is detected by means of Eriochrome Black T indicator, which has a red color in the presence of calcium and magnesium and a blue color when the cations are sequestered.

2.2 Hardness by Calculation: The sample is digested and analyzed by ICP. Hardness is then computed from results of the separate determinations of calcium and magnesium.

3.0 HEALTH AND SAFETY

See Standard Operating Procedure SWL-GA-111, "*Laboratory Safety Plan.*"

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

4.1 Cool to 4°C, HNO_3 to pH <2

4.2 Holding time is 6 months

5.0 INTERFERENCE AND POTENTIAL PROBLEMS

5.1 EDTA Titrimetric Method. Interferences from metal ions and suspended or colloidal organic matter are minimized through the procedure.

5.2 Calculation Method. Refer to section 5.0 in the current version of SOP SWL-IN-205.

6.0 EQUIPMENT/APPARATUS

6.1 EDTA Titrimetric Method. Standard laboratory titrimetric equipment.

6.2 Calculation Method. Refer to section 6.0 in the current version of SOP SWL-IN-205.

7.0 REAGENTS

7.1 EDTA titrimetric method.

7.1.1 Buffer Solution. Dissolve 16.9g NH_4Cl in 143ml conc. NH_4OH in a 250ml volumetric flask, add 1.25g of magnesium salt of EDTA and dilute to the mark with distilled water. Store in a tightly stoppered plastic bottle; stable for approximately one month. Discard when 1 or 2ml added to a sample fails to produce a pH of 10.0 + 0.1 at end point of titration.

If the magnesium salt of EDTA is unavailable, dissolve 1.179g disodium salt of ethylenediaminetetraacetic acid dihydrate (analytical reagent grade) and 780mg magnesium sulfate or 644mg magnesium chloride hexahydrate in 50ml distilled water. Add this solution to 16.9g NH_4Cl in 143ml conc. NH_4OH with mixing and dilute to 250ml with distilled water.

7.1.2 Indicator. Mix 0.5g Eriochrome Black T with 100g triethanolamine, dissolve in 100ml of 95% ethanol.

7.1.3 Standard EDTA titrant, 0.02N. Place 3.723g analytical reagent grade disodium EDTA in a liter volumetric flask and dilute to the mark with distilled water. Check with standard calcium solution by titration. Store in polyethylene. Check normality every month because of gradual deterioration.

7.1.4 Hydrochloric acid solution, 1:1

7.1.5 Methyl Red indicator. Dissolve 0.10g methyl red in distilled water in a 100ml volumetric flask and dilute to the mark.

7.1.6 Ammonium hydroxide solution, 3N. Dilute 210ml of conc. NH_4OH to 1 liter with distilled water.

7.1.7 Standard calcium solution, 0.02N (or 1000mg/L $CaCO_3$). Place 1.000g anhydrous calcium carbonate in a 500ml flask. Add a little at a time, 1 + 1 HCl until all of the $CACO_3$ has dissolved. Add 200ml distilled water. Boil for a few minutes to expel CO_2. Cool, add a few drops of methyl red indicator and adjust to intermediate orange color by adding 3N NH_4OH or 1:1 HCl as required. Quantitatively transfer to a 1 liter volumetric flask and dilute to the mark with distilled water. Standard will have a maximum shelf life of 6 months.

7.1.8 LCS/LCSD will be made from a standard that is independent of the standard calcium solution (section 7.1.7). This should have a value of 25mg/L — 80mg/L (titration), and 132mg/L (calculation method).

8.0 PROCEDURE

8.1 EDTA Standardization.

8.1.1 Place 10.0ml standard calcium solution in a flask containing 50ml distilled water. Add 1ml buffer solution & 1-2 drops of indicator.

8.1.2 Titrate slowly with continuous stirring until the last reddish tinge disappears. At end point the color is blue.

8.1.3 Calculate normality of EDTA

$$\text{N of EDTA} = \frac{0.2}{\text{mL of EDTA}}$$

8.1.4 Record results in the Standards Batch Form (see Attachment 1).

8.2 Hardness by EDTA titration with normal to high hardness.

8.2.1 The sample should require <15mL EDTA titrant and the titration should be completed within 5 minutes of buffer addition.

8.2.2 Place 25.0ml of sample into a 250ml Erlenmeyer flask. Add 1ml buffer solution and 1-2 drops of indicator.

8.2.3 Titrate slowly with continuous stirring with standard EDTA titrant until last reddish tint disappears, solution is normally blue at end point.

8.3 Hardness by EDTA titration with low hardness <5mg/L.

8.3.1 Use a larger volume of sample (100ml). Follow procedure in 8.2.

8.3.2 Use proportionately larger amounts of buffer & indicator. Follow procedure in 8.2.

8.4 If there is interference by metal ions causing fading or indistinct endpoints (for the EDTA titration method), then dilution of the sample should correct this problem. The analyst should note this interference in the comments section of the Analysis Log (see Attachment 2).

If available, hardness calculation from the ICP analysis should be performed to confirm the titration results. If the answers do not agree within 10% RPD a Non-Conformance Report (see SWL-GA-105) should be filled out noting this difference for the project officer.

8.5 Hardness by calculation. Refer to section 8.0 in the current version of SOP SWL-IN-205.

8.6 Write Up and Review Procedure

8.6.1 Fill out the appropriate information on the Standards Batch Form (see Attachment 1). This form only applies to the EDTA titrimetric method.

8.6.2 Fill out the appropriate information on the Analysis Log (see Attachment 2).

8.6.3 Review data in accordance with section 10.0 and SWL-IN-207.

8.6.4 Enter results into LIMS and place a copy of the Analysis Log with the LIMS episode worksheet.

9.0 CALCULATIONS

9.1 Hardness by the EDTA titrimetric method:

$$\text{Hardness as mg } \frac{CaCO_3}{L} = \frac{(A-B) \times C \times 50{,}000}{\text{mL Sample}} \times \text{Dilution}$$

Where:
A = Final reading of the EDTA titrant (ml)
B = Initial reading of the EDTA titrant (ml)
C = Normality of EDTA titrant

9.2 Hardness by Calculation

$$\text{Hardness, by equivalent } \frac{CaCO_3}{L} = 2.497\,(\text{Ca, mg/L}) + 4.118(\text{Mg, mg/L})$$

10.0 QUALITY ASSURANCE/QUALITY CONTROL

10.1 Analyze a method blank (PBW) with each sample batch.

10.2 Analyze a Laboratory Control Sample (LCW) and a Laboratory Control Sample Duplicate (LDW) with each sample batch.

10.3 Analyze a duplicate sample with each sample batch. If a client requires a ms/msd a duplicate sample is not required.

10.4 Analyze a matrix spike/matrix spike duplicate when requested by Client. Spike each 25ml sample with 2.0ml (8ml for a 100ml sample) of the standard calcium solution (section 7.1.7). This will give a spiking value of 80mg/L.

10.5 Analyze one standard at the detection limit (PQL) with each batch of samples.

11.0 WASTE DISPOSAL/POLLUTION PREVENTION

Refer to SOP SWL-GA-114, "*Hazardous Waste Management Plan.*"

12.0 METHOD PERFORMANCE

12.1 PBW will have a control limit of less than the reporting limit (as stated in section 12.4).

12.2 LCW/LDW will have control limits as stated in the Blue Book. If in-house control limits are not available, then use the control limit of 90-110% R and 20%RPD.

12.3 Duplicates will have a control limit of 20% RPD.

12.4 Reporting Limits

12.4.1 EDTA titrimetric method (IN140): 1.0mg $CaCO_3$/L

12.4.2 Calculation Method (IN145): 3.0mg $CaCO_3$/L

13.0 REFERENCES

13.1 Standards Methods for the Examination of Water and Wastewater, 19th ed., Method 2340B (calculation method) and 2340C (EDTA Titrimetric Method).

13.2 EPA Water and Waste Methods, Method 130.2 (EDTA titrimetric Method).

14.0 DEFINITIONS

Blue Book: .....A compilation of SWL in-house control limits

LCW/LDW: .....(Laboratory Control Sample/Laboratory Control Duplicate). Spiked method blank samples to provide laboratory accuracy and precision.

PBW: .....(Preparation Blank). A method blank sample to provide information on possible laboratory contamination.

Duplicate Sample: .....Done to provide precision information on the sample matrix.

MS/MSD: .....(Matrix Spike/Matrix Spike Duplicate). Samples that are spiked with a known amount to provide precision and accuracy information on the sample matrix.

15.0 ATTACHMENTS

15.1 Attachment 1: Hardness Standard Batch Form

15.2 Attachment 2: Hardness Analysis Batch Log

ATTACHMENT 1
Hardness Standard Batch Form

Hardness Standard Batch Form, SWL-IN-502

Standard Batch Log (502-ATT1.DOC) SWLO SOP rev. #_____

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

REAGENTS FOR HARDNESS

REAGENT	*LOT#*
1) NH_4CL	_____
2) NH_4OH	_____
3) Magnesium salt of EDTA	_____
4) Disodium salt of ethylenediaminetetraacetic acid dihydrate	_____
5) Eriochrome Black T	_____
6) Triethanolamine	_____
7) Ethanol	_____
8) Hydrochorlic Acid	_____
9) Methyl Red	_____
10) Anhydrous Calcium Carbonate	_____

STANDARD PREPARATION

Buffer Solution (Section 7.1.1):

16.9g (1) / 143 ml conc. (2) / 1.25g (3) ==========→ 250 ml (stable for 1 month) Analyst/Date:_____

Standard EDTA Titrant

3.723g (4) ==========→ 1000 ml (Shelf Life 6 months) Analyst/Date:_____

Standard Calcium Solution: SWL unique ID #:_____

Note: The above solutions were brought to volume with DI water.

EDTA STANDARDIZATION

Using 10.0 ml of Standard Calcium Solution in a flask containing 50 ml distilled water. Add 1 ml buffer solution and 1-2 drops of indicatior. Titrate with EDTA.

ml of EDTA, Titration #1 = _____ Titration #2 = _____ Titration #3 = _____

Avg of Titration #1, #2, and #3 = _____

N of EDTA = (0.2 / avg ml of EDTA) = _____ N Analyst/Date:_____

Southwest Laboratory of Oklahoma, Inc.

Verified By:_____

ATTACHMENT 2
Hardness Analysis Batch Log

HARDNESS ANALYSIS BATCH LOG

INORGANICS DEPARTMENT

HARDNESS ANALYSIS BATCH LOG (IN502A2.DOC) SWL-IN-502, REV. 4.0 Book #: IN-502-$CACO_3$-_____ Page: _____

Method reference: SM2340C / EPA 130.2

Analyst: _____ Date: _____ Prep Batch ID: _____ Standard Batch ID: _____ N of EDTA: _____

Test Code: _____ Calculation: Titration Method (IN140), mg/l = [(A-B) x C x 1000) / F Calculation Method (IN145), mg/l = (2.497 x D) + (4.118 x E)

#	SWL ID #	Smpl. Vol. (ml) (F)	Dil.	Initial EDTA (B) Vol. (ml)	Final EDTA (A) Vol. (ml)	ICP QC Reference #	Calcium (mg/l) (D)	Magnesium (mg/l) (E)	Final Result (mg/l)	Comments
1										
2										
3										
4										
5										
6										
7										
8										
9										
10										
11										
12										
13										
14										
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20										

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These SOPs will be followed until superceded by Pace Analytical Services SOPs.

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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. 5.1 — 07/08/02

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APPROVALS	DATES
Laura L Brown Procedure Prepared By	7-10-02 Date
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Chuck Hoover Laboratory Safety Officer	7-15-02 Date
Chuck Hoover Laboratory QA/QC Officer	7-15-02 Date

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

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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 be taken 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 an 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 seal 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 as 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

$$\text{Total Volatile Solids (mg/l)} = \frac{(A - B) \times 1000}{C}$$

Where: A = Weight of evaporating dish + residue before ignition in mg
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

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.

Sample Batch: .....Consists of a maximum of twenty samples per matrix.

Blank: .....Method Blank, consisting of 100ml of distilled water taken through the analysis to check for contamination.

Laboratory Control Sample: A sample of known concentration taken through the analysis to check the method accuracy.

Sample Duplicate: .....A sample taken through the analysis to check the matrix effect on method precision.

15.0 ATTACHMENTS

Attachment 1: TSS/TDS Logbook

Attachment 2: TS/VS/SS Logbook

ATTACHMENT 1
TSS/TDS Logbook

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

TM

TSS/TDS Log Book

INORGANIC DEPARTMENT

Book ID: IN-600-TSSTDSLog-14
Log Book START to FINISH DATE:

TSS/TDS Logbook, Ref: SWL-IN-600

INORGANIC DEPARTMEN

TSS/TDS Logbook (IN600A1.doc) SOP SWL-IN-600, Rev: 5.0 07/11/01
Method References: EPA 160.1/160.2/160.3, SM 2540B/2540C/2540D

Book # IN-600-TSSTDSLog-_

Test Code: _____ **Date of Analysis:** _____ **Time:** _____ **Batch ID:** _____
Date/Time In: _____ **Date/Time Out:** _____ **Temp. In:** _____ **Temp. Out:** _____ **Analyst's Initials:** ____

Dish or Crucible #	Sample ID	Comments	B Dish or Crucible Weight	C Sample Volume	A Dish or Crucible + Residue 1st	A Dish or Crucible + Residue Final	D Dish or Crucible + Residue 1st	D Dish or Crucible + Residue Final	A-B A-D D-B	Dilution	Final Results	Units	% Rec % RPI

TDS: 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

TSS: Total Suspended Solids, mg/L = $\frac{(A-B) \times 1000}{C}$

Where: A = Weight of crucible dish + residue in mg.
B = Weight of crucible dish in mg
C = mL of sample used

ATTACHMENT 2
TS/VS/SS Logbook

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

TM

TS/VS/SS Log Book

INORGANIC DEPARTMENT

Book ID: IN-600/609-TSVSSSLog-____
Log Book START to FINISH DATE:

Ref: SWL-IN-600/SWL-IN-609
In600a2cov

TS/VS/SS Logbook, Ref: SWL-IN-600 / SWL-IN-609

INORGANIC
DEPARTMEN

TS/VS/SS Logbook (IN600A2.doc or IN609A1) SOP SWL-IN-600, Rev: 5.0 07/11/01 SOP SWL-IN-609, Rev: 2.0 07/11/01

Method References: EPA 160.5/SM2540-F/SM 2540B/2540C/2540D/2540E/EPA 160.1/160.2/160.3/160.4 Book # IN-600/609-TSVSSSLog- ____

Test Code: _____ **Date of Analysis:** _____ **Time:** _____ **Batch ID:** _____

Date/Time In: _____ **Date/Time Out:** _____ **Temp. In:** _____ **Temp. Out:** _____ **Analyst's Initials:** ____

Dish or Crucible #	Sample ID	Comments	B Dish or Crucible Weight	C Sample Volume	A Dish or Crucible + Residue 1st	A Dish or Crucible + Residue Final	D Dish or Crucible + Residue 1st	D Dish or Crucible + Residue Final	A-B A-D D-B	Dilution	Final Results	Units	% Rec % RPD

TS: 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

VS: Total Volatile Solids, mg/L = $\frac{(A-B) \times 1000}{C}$

Where: B = Weight of evaporating dish in mg after ignition
C = mL of sample used

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OV-103 / -303

The Standard Operating Procedures (SOPs) contained herein are adopted by Pace Analytical Services.

These SOPs will be followed until superceded by Pace Analytical Services SOPs.

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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

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APPROVALS	DATES
James Whittaker	10/11/00
Procedure Prepared By	Date
Harry M Bray	10-24-00
Program Manager or Section Supervisor	Date
Bryan Hach	10/24/2000
Laboratory Safety Officer	Date
Chuck Hoover	10/24/00
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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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

15.0 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

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

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

Typist: LLR

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. 6.3 — 07/23/03

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APPROVALS	DATES
Melissa Chamick Procedure Prepared By	8-12-03 Date
James Whittaker Program Manager or Section Supervisor	8/12/03 Date
Chuck Jones Laboratory Safety Officer	7/30/03 Date
Chuck Jones Laboratory QA/QC Officer	7/30/03 Date

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

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ANALYSIS OF VOLATILE ORGANICS BY GC/MS (SW846 METHOD 8260B)

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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 5mL 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.

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.

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. A single solution containing internal standards and surrogate standards are prepared containing the above at a concentration of 25ug/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.

8.4.1.1 Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan no more than 20 scans prior to the elution of BFB. Do not background subtract part of the BFB peak.

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 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 spanning retention time range of analytes of interest.

8.5.2 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 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 linear regression does not work (the coefficient of determination, r^2 ,is less than 0.99), the system will be recalibrated if the compound is detected in the samples. In those instances when the %RSD is greater than 15% and linear regression is less than 0.99; the five-point, initial calibration, has demonstrated that the compound could have been detected in the sample had it been present, at or above the low level standard.

8.5.4.3 If the % RSD of any compound is greater than 15%, the initial calibration may be used if the average of the %RSD values for **all** analytes in the calibration is less than or equal to 15%. The average %RSD is calculated by summing the RSD values for each analyte and dividing by the total number of analytes in the calibration. If no analyte has an RSD above 15%, then the average RSD does not need to be calculated.

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 percent difference from the expected values should be less than 15% if any individual analyte exceeds 15% difference, the average of all target analytes may be used. The average percent difference for all target analytes must be less than 15%.

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/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	50	5
10	4	4	4	50	20
10	10	10	10	50	50
10	20	20	20	50	100
10	40	40	40	50	200

Soil-5mL Purge/Precept or 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)
8	8	8	50	5
32	32	32	50	20
80	80	80	50	50
160	160	160	50	100
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 bef

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: 50ppb. 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.

The calibration may be verified if the average of the % differences and/or % drifts for **all** analytes in the calibration is less than or equal to 15%. The individual % difference/drift for each compound should be less than 50%, and must have a %Difference/Drift less than 60%. If the % Difference/Drift is greater than 60%, 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. Before proceeding with sample analysis verify that all other client specific or contractually required calibration verification requirements have been satisfied.

8.6.2 The calibration verification is performed after the BFB and must meet all criteria before continuing analysis. 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 5mL. 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, 10µL of the surrogate Spiking Solution and 10µL of the Internal Standard Spiking Solution are added through the valve bore of the syringe, which is equivalent to a concentration of 50µg/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.

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.

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 (Un Enhanced & Enhanced) & compared to NIST Library Spectra, excluding internal standards and surrogates

8.12.5 Tentatively Identified Compounds (TICs)

8.12.6 Internal Standards/Surrogates

8.13 Manual Integration: Manual integration must only be used to correct incomplete or incorrect integration performed by the data system. Manual integration must not be

performed as a means to satisfy QC acceptance criteria. All manual integration must be performed in a consistent manner.

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

See Standard Operating Procedure SWL-GA-114, "*Hazardous Waste Management Plan.*"

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"*.

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
Chloroethane	64	66
2-Chloroethylvinyl Ether	63	43, 43
Chloroform	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			

Table 6
Volatile Calibration Check Compounds (CCC)

- 1,1-Dichloroethene
- Chloroform
- 1,2-Dichloropropane
- Toluene
- Ethylbenzene
- Vinyl Chloride

Initial Calibration: 5,20,50,100,200ppb (soil/5mL purge)
Max%RSD: 30% using first order regression above 15%
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

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: $\% \text{ Surrogate Recovery} = \frac{Qd \times 100\%}{Qa}$

Where:
Qd = Quantity determined by analysis
Qa = Quantity added to sample

Matrix Spike recovery and RPD:

$$\text{Matrix Spike Recovery} = \frac{SSR - SR \times 100}{SA} \qquad \text{Matrix Spike RPD} = \frac{|MSR - MSDR|}{(½)(MSR + MSDR)} \times 100$$

Where:
SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

Laboratory Control Spike (LCS) Recovery: $\text{LCS Spike Recovery} = \frac{SSR - SR \times 100}{SA}$

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.

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

ATTACHMENT 2
Encore Sample Preservation Form

ATTACHMENT 3
VOA Vial Preservation/Preparation Form

APPENDIX 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

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 $NaHSO_4$ 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.

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Standard Operating Procedure

Analysis of Volatile Organics by GC/MS 8260B — 25mL Purge

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APPROVALS	DATES
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ANALYSIS OF VOLATILE ORGANICS BY GC/MS (8260A-25mL PURGE)

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1.0 SCOPE AND APPLICATION

1.1 Method 8260B, 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.

1.3 There are no deviations from the method (8260A) in this procedure.

2.0 METHOD SUMMARY

2.1 An inert gas, helium, is bubbled through an aqueous 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, 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 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

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, notably methylene chloride, acetone, and chloroform, present in the air of most analytical laboratories, 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 EQUIPMENT / APPARATUS

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. 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

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 an 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 9. The criteria the initial calibration must meet to be acceptable are 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).

8.5.4.3 If the % RSD of any compound is greater than 15%, the initial calibration may be used if the average of the % RSD values for **all** analytes in the calibration is less than or equal to 15%. The average % RSD is calculated by summing the RSD values for each analyte and dividing by the total number of analytes in the calibration. If no analyte has an RSD above 15%, then the average RSD does not need to be calculated.

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	Gases	8260 Extra Cmpd.	Int. Std.	Standard	Acrolein/Acrylonitrile

Stock Std. (µL)	Stock Std. (µL)	Stocks Standard (µL)	Final Conc. (ng/mL)	Final Conc. (ng/mL)	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 37°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. 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 calibration verification must meet to be acceptable are also found in Table 6.

The calibration may be verified if the average of the % differences and/or % drifts for **all** analytes in the calibration is less than or equal to 15%. The individual % difference/drift for each compound should be less than 50%, and must have a %Difference/Drift less than 60%. If the % Difference/Drift is greater than 60%, 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. Before proceeding with sample analysis verify that all other client specific or contractually required calibration verification requirements have been satisfied.

All other compounds should have a %Drift less than 40%, except for the following which should have %Drift less than 80%: gases, 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. For 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.

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 compounds, excluding internal standards and surrogates

8.10.5 Tentatively Identified Compounds (TICs)

8.10.6 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 25 mL of filtered tap water spiked to yield a concentration of 10 ppb internal standard and surrogate, or a 40mL volatile vial of filtered tap water loaded onto 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). Exceptions can be made, however, for the common laboratory contaminants acetone, chloroform, and methylene chloride. These compounds must be present at concentrations less than 5 times the practical quantitation limit (PQL), or 5 ppb, unless otherwise requested by the client.

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 and Laboratory Control Spike Duplicate (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. However, an LCS or LCSD will be considered acceptable if no more than 10 percent of the spiked target compounds fall outside their recovery limits. Limits are derived using a control chart program as per SOP SWL-GA-112, "*Control Chart.*"

10.2.3 If more than 10 percents of 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 SOP SWL-GA-114, "*Hazardous Waste Management Plan.*"

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 SW846-8260B (Revision 2 – December, 1996)

13.2 *Control Charts* — SOP SWL-GA-112

13.3 *Definition and Determination of Detection Limits* — SOP SWL-GA-113

13.4 *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 server-based processing system. Data is archived from the server weekly.

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)	Analyte	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Acetone	43	58	Trans-1,2-Dichloroethene	96	61, 98
Acrolein	56	55	1,2-Dichloropropane	63	112
Acrylonitrile	53	52, 51	1,3-Dichloropropane	76	78
Benzene	78	—	2,2-Dichloropropane	77	97
Bromobenzene	156	77, 158	1,1-Dichloropropene	75	110, 77
Bromochloromethane	128	49, 130	Ethylbenzene	91	106
Bromodichloromethane	83	85, 127	2-Hexanone	43	58,57
Bromoform	173	175, 254	Hexachlorbutadiene	225	223, 227
Bromomethane	94	96	Isopropylbenzene	105	120
2-Butanone	43	72, 57	p-Isopropyltoluene	119	134, 91
n-Butylbenzene	91	92, 134	Methylene Chloride	84	86, 49
sec-Butylbenzene	105	134	4-Methyl-2-Pentanone	43	58
tert-Butylbenzene	119	91, 134	Naphthalene	129	—
Carbon Disulfide	76	78	n-Propylbenzne	91	120
Carbon Tetrachloride	117	119	Styrene	104	78
Chlorobenzene	112	77, 114	1,1,1,2-Tetrachloroethane	131	133, 119
Chloroethane	64	66	1,1,2,2-Tetrachloroethane	83	131, 85
2-Chloroethylvinyl Ether	63	43, 43	Tetrachloroethene	166	168, 129
Chloroform	83	85	Toluene	92	91
Chloromethane	50	52	Trans-1,3-Dichlorpropene	75	77
2-Chlorotoluene	91	126	1,2,3-Trichlorobenzene	180	182, 145
4-Chlorotoluene	91	126	1,2,4-Trichlorobenzene	180	182, 145
cis-1,3-Dichloropropene	75	77	1,1,1-Trichloroethane	97	99, 61
1,2-Dibromo-3-Chloropropane	75	155, 157	1,1,2-Trichlorethane	83	97, 85
Dibromochloromethane	129	127	Trichloethene	95	130, 132
1,2-Dibromoethane	107	109, 188	Trichlorofluoromethane	101	103
Dibromomethane	93	95, 174	1,2,3-Trichloropropane	75	77
1,2-Dichlorobenzene	146	111, 148	1,2,4-Trimethylbenzene	105	120
1,3-Dichlorobenzene	146	111, 148	1,3,5-Trimethylbenzene	105	120
1,4-Dichlorobenzene	146	111, 148	Vinyl Chloride	62	64
Dichlorodifluoromethane	85	87	o-Xylene	106	91
1,1-Dichloroethane	63	65, 83	m,p-Xylene	106	91
1,2-Dichloroethane	62	98	Vinyl Acetate	43	86
1,1-Dichloroethene	96	61, 63			
cis-1,2-Dichloroethene	96	61, 98			

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	• Chloroform
• 1,2-Dichloropropane	• Toluene
• Ethylbenzene	• 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	• 1,1,2,2-Tetrachloroethane
• 1,1-Dichloroethane	• Chlorobenzene
• Bromoform	

Minimum Relative Response Factor (RRF)	0.100 for Chloromethane
	1,1-Dichloroethane
25mL Purge RRF:	0.300 for Chlorobenzene

Table 7
Compound List and Method Reporting Limits

Volatile Compound	CAS#	25mLWater	Volatile Compound	CAS#	25mL Water
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 = pike 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

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RD-113 / -124 / -138

The Standard Operating Procedures (SOPs) contained herein are adopted by Pace Analytical Services.

These SOPs will be followed until superceded by Pace Analytical Services SOPs.

Signature:	Signature:	Signature:
Chuck Hoover	Roger C. Splinter	Dave Neal
Quality Assurance Officer	Dir. of Quality, Safety, Training	General Manager
Pace Analytical Services, Inc.	Pace Analytical Services, Inc.	Pace Analytical Services, Inc.
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Standard Operating Procedure

Quality Control for Radiochemical Analyses

Document No.: SWL-RD-113

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QUALITY CONTROL FOR RADIOCHEMICAL ANALYSES

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Instrumentation QC Requirements 4

1.0 SCOPE AND APPLICATION

The purpose of this procedure is to provide quality control requirements for radiochemical analyses when not specified by the client.

2.0 METHOD SUMMARY

This procedure gives minimum internal QC acceptance criteria for all radiochemical analyses.

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

Not applicable.

7.0 REAGENTS

Not applicable.

8.0 PROCEDURE

8.1 Analyses:

8.1.1 As per standard operating procedures, all analyses have certain QA/QC requirements that must be met before results can be reported.

8.1.2 QC requirements listed below shall be met before samples can be reported to clients or as per client specific requirements.

8.1.3 Standards (LCS):

Standard Result = 75% to 125% of known

8.1.4 Blanks (BLK):

Blank Result = Within error estimate window of minimum detectable activity for that analysis.

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8.1.5 Duplicates (DUP):

$$|\text{Result} - \text{Dup. Results}| \ / \ [(SE)^2 + (DE)^2]^{.5} < 1.5$$

Where: SE = The error estimate of the Sample Result
DE = The error estimate of the Duplicate Sample Result

8.1.6 Matrix Spike (MS), Matrix Spike Dup. (MSD):

Spike Recovery *:
NMSR - NSR / SPIKE = 75% to 125% of known
NMSDR - NSR / SPIKE = 75% to 125% of known

Precision *:

$$|\text{Result} - \text{Dup. Result}| \ / \ [(SE)^2 + (DE)^2]^{.5} < 1.5$$

Where:
NMSR = Normalized Matrix Spike Result
NSR = Normalized Sample Result
NMSDR = Normalized Matrix Spike Dup. Result
SPIKE = Known spike activity added
SE = The error estimate of the Normalized Sample Result
DE = The error estimate of the Normalized Dup. Sample Result

NOTE: In the case of matrix spike and matrix spike duplicates for gross alpha beta analyses, these calculations are not used as a criteria of acceptance due to the large variance in sample matrixes that interfere with this analysis and are analyzed to demonstrate the presence of that interference or lack thereof.

8.1.7 In addition to minimum acceptable criteria requirements for each analyses, Standards and Blanks will be plotted on a Shewhart Control Chart for each radionuclide to determine possible trends that would effect overall integrity of each analysis. In addition, matrix spike recoveries will be plotted on all analyses that do not include the use of carrier or internal tracer recovery determinations. Other indicators such as duplicates, replicates, spikes, etc. are not plotted because they would not necessarily be accurate indicators of adverse quality trends. If a trend is noticed, immediate action should be taken to find cause so as to maintain a high degree of reliability of QC and sample data. After detection of a problem indicated by the control charts, stop performing analyses, make corrections as needed, and run a QC batch without samples to check the procedure. If the parameters return to normal, continue sample analyses; if not, make correction and test again until analyses falls back into historical acceptance range as determined by the control charts.

8.1.8 Reanalyze all samples that do not meet requirements from Attachment 1.

8.1.9 Minimum QC frequency requirements are listed in Attachment 1.

8.2 Instrumentation:

8.2.1 The continuing overall performance of each instrument used should be monitored using control charts with Acceptance Criteria met to insure overall sample analyses quality.

8.2.2 Using Shewhart Control Charts plot parameters in Attachment 1 and maintain acceptable operating conditions. If any parameter falls out of the acceptable range stop using that particular instrument and determine corrective action. Instruments are not to be used until all parameters meet minimum requirements and should be labeled out of service.

8.2.3 Recalibrate all instruments at least annually, unless specified differently in the individual SOP. See Attachment 1 for frequency of quality point checks.

8.2.4 Maintain instrument backgrounds within ± the error of the analysis minimum detectable activity required for each analysis.

9.0 CALCULATIONS

9.1 As stated in "Procedure" section.

9.1.1 All negative activities shall be reported as such. If the sum of the activity and the measurement uncertainty at 3-sigma is a negative number, the cause shall be investigated and evaluated to determine if it is systematic or random. If the cause is systematic, it shall be corrected. If the cause is random it shall be documented in the Case Narrative. Recurrent problems with significant negative results suggest that the background subtraction and/or blank subtraction, if applicable, are in error or that the estimate of error is low. Investigation of such problems and documentation of the resolution is required and shall be discussed in the Case Narrative.

10.0 QUALITY ASSURANCE/QUALITY CONTROL

As stated herein.

11.0 WASTE DISPOSAL/POLLUTION PREVENTION

Not applicable.

12.0 METHOD PERFORMANCE

Not applicable.

13.0 REFERENCES

13.1 SWLO Standard Operating Procedures, G-Series, and R-Series as indicated.

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13.2 Prescribed Procedures for Measurement of Radioactivity in Drinking Water, EPA-600/4-80-032, August, 1980, EPA.

14.0 DEFINITIONS

Not applicable.

15.0 ATTACHMENTS

Attachment 1: Radiochemical QC Requirements, Instrumentation QC Requirements

ATTACHMENT 1

RADIOCHEMICAL QC REQUIREMENTS

SOP	ANALYSES	DUP *	MS/MSD **	Standard *	Blank *	Carrier / Tracer Recovery MIN	Carrier / Tracer Recovery MAX
SWL-RD-101	Gross Alpha Beta	X	X	X	X	N/A	N/A
SWL-RD-102	Total Radium	X		X	X	50%	110%
SWL-RD-103	Radium-226	X		X	X	50%	110%
SWL-RD-104	Radium-228	X		X	X	35%	110%
SWL-RD-105	Radium-226	X		X	X	50%	110%
	Radium-228	X		X	X	35%	110%
SWL-RD-106	Strontium	X		X	X	35%	110%
SWL-RD-107, SWL-RD-120	Isotopic Thorium	X		X	X	20%	110%
SWL-RD-108, SWL-RD-121	Isotopic Uranium	X		X	X	20%	110%
SWL-RD-109	Radon 222	X			X	N/A	N/A
SWL-RD-110	Lead 210	X		X	X	20%	110%
SWL-RD-129	Tritium	X	X	X	X	N/A	N/A
SWL-RD-130, SWL-RD-120, SWL-RD-121	Americium	X		X	X	20%	110%
	Plutonium	X		X	X	20%	110%
	Curium	X		X	X	20%	110%
	Neptunium	X		X	X	20%	110%
SWL-RD-112	Polonium 210		X	X	X	N/A	N/A
SWL-RD-118	Gamma Spec.	X		X	X	N/A	N/A

* One per batch of 1 to 20 samples.
** One MS and MSD per batch of 1 to 20 samples.
(1) Client request only.

INSTRUMENTATION QC REQUIREMENTS

SOP	INSTRUMENT	FWHM	SRCE CHECK	BKG	SRCE CHECK FREQUENCY	BKG Determination	BKG CHK
SWL-RD-119	Alpha Spectrometers	X	X	X	weekly	monthly	monthly
SWL-RD-117	Gas Prop. Cntr.		X	X	daily/with use	weekly/with use	daily/with use
SWL-RD-123	Ludlum Light Scint.		X	X	daily/with use	daily/with use	daily/with use
SWL-RD-125	Gamma Spec.	X	X	X	daily/with use	monthly/with use	monthly/with use
SWL-RD-131	Packard L. S. U.		X	X	daily/with use	daily/with use	daily/with use

Typist: SRM

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Standard Operating Procedure

The Preparation and Tracking of Standards, Tracers, and Reagents

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THE PREPARATION AND TRACKING OF STANDARDS, TRACERS, AND REAGENTS

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1.0 SCOPE AND APPLICATION

1.1 This procedure gives instructions for the preparation and traceability of radioactive isotopes and reagents in the laboratory.

1.2 This procedure applies to sources and standards for instrument calibration and for the preparation of stock solutions (initial standard dilutions) and working solutions (subsequent dilutions of stock solutions) to be used in the analysis of radiochemical analyses.

2.0 METHOD SUMMARY

New radioactive tracers or standards are received and assigned a log identification number and recorded in the Isotope Log. After noting holding times, and calculating volume needed, isotopes are diluted into a "Stock Solution". Subsequent dilutions from the stock solution are termed and used as "Working Solutions". All dilutions are assigned a unique number and are recorded in the Isotope Log. Each dilution's activity is calculated and maintained in a binder divided into isotope sections, which is used for reference when standard recoveries are analyzed. All sources and standards for instrument calibration are also logged into the Isotope Log and all certification pages are kept in this same binder.

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 Due to the high activities associated with standards and tracers, extreme care should be given to insure that no contamination exists after preparation. Swipe surveys should be conducted after preparations are completed.

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.

6.2 Glass bottles with screw caps.

6.3 Class "A" Volumetric pipettes.

7.0 REAGENTS

7.1 Reagent grade chemicals for dilutions.

7.2 NBS traceable certified sources, standards and tracers.

8.0 PROCEDURE

8.1 The radiochemistry manager or designee is responsible for the dilutions and recording of isotope information for all radioactive tracers and standards used in the radiochemistry laboratory. Standards, sources, and tracers are locked in clearly labeled storage cabinet with the managers only having access.

8.2 Receive standards from approved vendors. Assure that standards being received have a maximum error of 6% at the 99% confidence level (total error) and that analyte impurities are no greater than 0.5% of the tracer activity. Also assure that the standard is within reasonable limits of the expiration date and has the correct curie content requested in the purchase order. If any of the above criteria are not met, contact the supervisor and quality assurance officer followed by the completion of a corrective action report. Date and initial calibration page. Make copies of calibration page and give to QA/QC Officer. All standard and tracers used in analysis of samples shall be NIST traceable. If standards or tracers cannot be obtained that are NIST traceable, it shall be noted on the report.

8.3 All standards and reagents shall be clearly labeled at the bench. At a minimum, each label shall include the standard and/or reagent name, laboratory identification number, prepared date, initials of preparing individual, and expiration date.

8.4 Assign an identification number to new undiluted standard and sources, and record in Isotope Log as below:

Isotope-Number-Code
XX-XXXX-XX
EX: Pu-0001-VS

CODES:
CS = "Calibration Sources"
VS = "Vendor Solution"
SS = "Stock Solution"
WS = "Working Solution"

Four digit number runs sequentially from 0001 to 9999.

8.5 Stock Solutions

8.5.1 Calculate volume necessary to dilute standard from vendor to approximately 1000 dpm/ml using the formula:

$$Vo \times Ao = Vn \times An$$

Where: Vo =volume or weight or original — Ao =activity of original (dpm/ml)
Vn =volume of diluted standard — An =activity of diluted standard (dpm/ml)

8.5.2 If the original activity as reported by the supplier, was in activity per unit weight continue with the next step. If the original activity as reported by the supplier wa

in activity per unit volume use a class "A" pipette to transfer standard to a volumetric flask, and continue with step 8.5.6.

8.5.3 Using an analytical balance first weigh a screw cap volumetric flask to be used for the dilution and record the tare weight.

8.5.4 Using a disposable dropper remove the standard from the capsule (being careful not to spill any solution on the counter top) and dispense it into the weighed volumetric flask.

8.5.5 Weight and record the gross weight of the volumetric flask with the standard in it.

8.5.6 Add Acid to the volumetric flask (per size determined in step 1) at the rate of 5mls/100mls of final solution. The same acid should be used as was used by the supplier for the original sample matrix. (Do not acidify Tritium standards).

8.5.7 Assign a new identification number using the same syntax as in step 8.3. The suffix is "SS" for this stock solution.

8.5.8 Label the flask identification number assigned and store in the locked standards storage cabinet.

8.5.9 Record results in the Isotope Log and on the Isotope Standard Dilution Form. Place this information along with a copy of standard certificate in the Standard Records/Decay Binder.

8.6 Working Solutions

8.6.1 From the stock solutions calculate what dilution is necessary to prepare standards/tracers to meet contract required spiking levels by the formula:

$$Vs \times As = Vw \times Aw$$

Where:
Vs = volume of stock solution
As = activity of stock solution dpm/ml
Vw = volume of working solution
Aw = activity of working solution dpm/ml

8.6.2 Using a class "A" pipette and a Class "A" volumetric flask carefully pipette the volume of stock solution calculated in step 8.6.1 into the volumetric flask.

8.6.3 Add acid to the volumetric flask at the rate of 5mls/100mls of final solution. The same acid should be used as was used in the stock solution.

8.6.4 Dilute to volume with D.I. water.

8.6.5 Assign a new identification number using the same syntax as in step 8.3. The suffix is "WS" for this working solution.

8.6.6 Label the flask with the identification number assigned and store in the locked standards storage cabinet.

8.6.7 Record results in the Isotope Log and on the Isotope Standard Dilution Form. Place this information in the Standards Records/Decay Binder.

8.6.8 Only use working solutions for spiking standards and tracers. This prevents contamination of stock solution and traceability of possible dilution problems. Also, so as to minimize possible contamination, make working solution volumes as small as possible.

8.7 Standard Checks and Verification

8.7.1 After working solutions are made, check accuracy of calculated activity by comparing a previously verified solution of the same isotope. Each isotope is compared differently due to the nature of the chemical separation and counting process used for that isotope.

8.7.2 Standards and tracers shall be verified prior to initial use. Three verification measurements of a standard shall be used to determine the mean value and standard deviation of the verification results.

8.7.3 Place all information in the Standards Records/Decay Binder. See below:

8.7.3.1 Alpha Spectroscopy and Beta Emitters: Standard and tracer "Standardization Verification" are carried out through the counting of three replicates of co-precipitations of the nuclide with another standardized isotope of the same element or carrier utilized in the analytical procedure and utilizing the second isotope or carrier to correct for chemical recovery.

8.7.3.2 Gross Alpha, Beta, or Gamma Spectroscopy Standard: Where not already standardized as above, gross alpha, beta, or gamma spectroscopy solutions can be standardized by direct counting. Gross alpha, beta requires the preparation of three replicate planchets (see SWL-RD-101) and direct counting for gross alpha, beta. The standardization of gamma emitters can be carried out by the direct counting of one-five hundred-milliliter sample, in a Marrinelli beaker three times for gamma spec.

8.7.3.3 Ra-226 Solutions: Ra-226 solutions will be standardized by placing a known volume of standard in each of three radon bubblers, performing a first de-emanation and after 4 days, a second de-emanation and then counting per (SWL-RD-103) in a Lucas Cell scintillation counter. Ra-226 solutions can also be standardized using the above gross alpha method.

8.7.4 Counting times and standard volumes should be selected to yield a 2 sigma counting error of 2%. The certificate value (not including any uncertainty) shall lie within the 95% confidence interval determined from the mean and two sigma standard deviation of the three measurements. The two sigma value used for the 95% confidence interval shall not exceed 10% of the mean value of the three verification measurements.

8.7.5 If standards do not compare favorably with previous standards as stated above, check all the data and dilution information recorded in the standard dilution form for accuracy. If the information is correct, re-test the standard.

If after the second test, the results are the same first test, notify the vender and send a portion of the stock dilution back to them for testing.

8.7.6 If the vender confirms erroneous activity, return all dilutions of the standard, and request a replacement. Make note of this on all dilution forms and in the Isotope Log. If the Vender refuses to replace the solution label the dilutions "DO NOT USE" and take out of use.

8.8 Standard and Tracer Decay Calculation.

8.8.1 Recalculate all tracers and standards according to the following schedule:

Half Life of Isotope Frequency
0 to 10 years recalculate activity each use.
11 to 50 years Recalculate activity 1 x per Quarter.
51 years and greater Recalculate 1 x per year.

8.8.2 To recalculate the amount of an isotope remaining, use the following formula:

$$\text{Decimal fraction remaining} = e^{-dt}$$

Where:

$d = \frac{.693}{T\,½}$

T1/2 =half life of isotope in question.
T = time elapsed from original activity to present.
(Note: that this time must be in the same units used T1/2).

8.8.3 Record results of decay corrections on the Isotope Standard Dilution Form.

8.8.4 Use this form to retrieve actual decayed activities of each standard or tracer when doing calculations.

8.9 Expiration of Standards and Tracers

8.9.1 Standards and tracer stock solutions shall be verified every five years. Subsequent working solutions shall be verified at least annually.

8.9.2 Any standard or tracers that are determined to be unusable for what ever reasons, (i.e. contamination, questionable dilutions, expiration) should be included in the radioactive waste stream and handled as per standard operating procedure. Make note on Isotope Standard Dilution Form.

8.9.3 Standards and Tracers shall be re-verified before the expiration date by following the receipt verification process as described in 8.7.1. The stock solution shall be verified by making all new subsequent dilutions. All subsequent solutions of a standard or tracer that has not been re-verified, and that is expired shall not be used and labeled as "Do Not Use".

8.10 Use the Isotope Log as the main reference and tracking log for all radioactive sources, tracers and standards in the lab. Place all supporting data and printouts with each Isotope Standard Dilution Form.

8.11 Tracers and standards used in analyses shall be from a separate source than the ones used for calibration of the counting instruments.

8.12 Selection and Level for LCS and tracers.

8.12.1 The LCS shall be of the same analyte as the sample analyte and shall be at least 2 times but not greater than 20 times the required minimum detectable activity (MDA) with the following exceptions.

8.12.1.1 For MDA's of low activity the analyte shall be at a level where the random counting error does not exceed 10% in the counting time required to attain the required MDA.

8.12.2 Tracers shall be at approximately equivalent activities as LCS's.

8.13 Reagent Preparation Log

8.13.1 Reagent ID# - Assign an identification number to new reagents that are prepared as per each analytical SOP, in the Reagent Preparation Log (attachment 3), following the syntax described below.

RS-XXXXX, where "RS" stands for Reagent Solution, and "XXXXX" is a continuing sequential number.

8.13.2 Reagent Lot # - Record the original reagent lot number as supplied by the manufacturer.

8.13.3 Expire Date - The reagent Expiration Date assigned shall use the earliest expiration date between the parent expiration date and the expiration date as stated in the individual SOP.

8.13.4 Parent Volume – The initial volume (or weight, if the parent is a crystal or powder) of reagent added to the total volume to achieve the desired concentrations.

8.13.5 Total Volume – The total amount of reagent prepared.

8.13.6 SOP #, REV#. – The SWL SOP number, and revision # of the SOP used to prepare the reagent.

8.13.7 When performing analyses that use these reagents, record on batch preparation sheets as described in the individual SOPs or supply a supplemental listing when it is not practicable, in the form of supplemental documentation, these prep batch records are stored as a part of the file for future reference if needed. Reagents are verified through reagent blank analyses.

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

- Kocher, David C., Radioactive Decay Data Tables, DOE/TIC-11026, 1981.

14.0 DEFINITIONS

Not applicable.

15.0 ATTACHMENTS

Attachment 1: "Isotope Log"

Attachment 2: "Isotope Standard Dilution Form"

Attachment 3: "Reagent Preparation Log"

ATTACHMENT 1
Isotope Log

Isotope Log, SWL-RD124

ISOTOPE LOG (124-ATT1.DOC)

Book # RD124-PRP-____- Page # - 1

* - Activity : as of referance date from manufacturer.

Verified: _____

Date	Int	Isotope	Log ID	Activity	Units	Phys. Form

Southwest Laboratory of Oklahoma, Broken Arrow, OK

ATTACHMENT 2
Isotope Dilution Form

Isotope Dilution Form SWL-RD124

ISOTOPE DILUTION FORM (124-ATT2.DOC)

ISOTOPE: _____ ID: _____

HALF LIFE: _____ DILUTION TYPE: _____

DILUTION DESCRIPTION:

ORIGINAL ID: _____ ORIGINAL ACTIVITY: _____

ORIG. ACTIVITY DATE: _____

DILUTION DESC: _____

PREP. DATE: _____

NEW SOLUTION: EXPIRATION DATE: _____

INITIAL	DATE	NEW ACTIVITY		INITIAL	DATE	NEW ACTIVITY

SOLUTION USED UP DATE : _____

EXPIRED WASTE: _____

ATTACHMENT 3
Reagent Preparation Log

1

Reagent Prep. Log, SWL-RD124

REAGENT PREP BOOK (124-ATT3.DOC) SWLO, SOP REV. # _____ Book # RD124-RPP-_1_

Verified ___

Reagent	Reagent ID #	Reagent Lot #	Expire Date	Parent Volume	Total Volume	SOP #, REV #

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SOUTHWEST LABORATORY OF OKLAHOMA, INC. *and*
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Standard Operating Procedure

Gamma Spectrometer Operation/Calibration

Document No.: SWL-RD-125

Rev No. / Date: Rev. No.: 1.0 — 05/02/01

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APPROVALS	DATES
Procedure Prepared By	Date 5/2/01
Program Manager or Section Supervisor	Date 5/2/01
Laboratory Safety Officer	Date 5/8/01
Laboratory QA/QC Officer	Date 5/8/01

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

Document Status

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[✓] Official Copy OC No.: 2611 Issued to: TTNUS Date: 10/15/03 QA: CHH

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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 SWL-GA-111, "*Laboratory Safety Plan.*"

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 Detection 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/POLLUTION PREVENTION

See Standard Operating Procedure SWL-GA-114, "*Hazardous Waste Management Plan.*"

12.0 METHOD PERFORMANCE

Not applicable.

13.0 REFERENCES

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

Gamma Spec Run Log, SWL-RD125

GAMMA SPEC RUN LOG (125-ATT2.DOC) Book # RD125-INST-_1_

Verified _____

Date	Analyst	Sample ID	Sample Volume	Geometry	Count Time	File ID

SOUTHWEST LABORATORY OF OKLAHOMA, INC. *and*
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Standard Operating Procedure

Glassware Cleaning, Radiochemical Analyses

Document No.: SWL-RD-138

Rev No. / Date: Rev. No.: 1.1 — 08/22/00

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APPROVALS	DATES
Procedure Prepared By	Date 8/22/00
Program Manager or Section Supervisor	Date 8/22/00
Laboratory Safety Officer	Date 8/28/00
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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

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RD-141

The Standard Operating Procedures (SOPs) contained herein are adopted by Pace Analytical Services.

These SOPs will be followed until superceded by Pace Analytical Services SOPs.

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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
Laboratory Safety Officer	Date 9/19/2000
Laboratory QA/QC Officer	Date 9/19/00

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

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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".

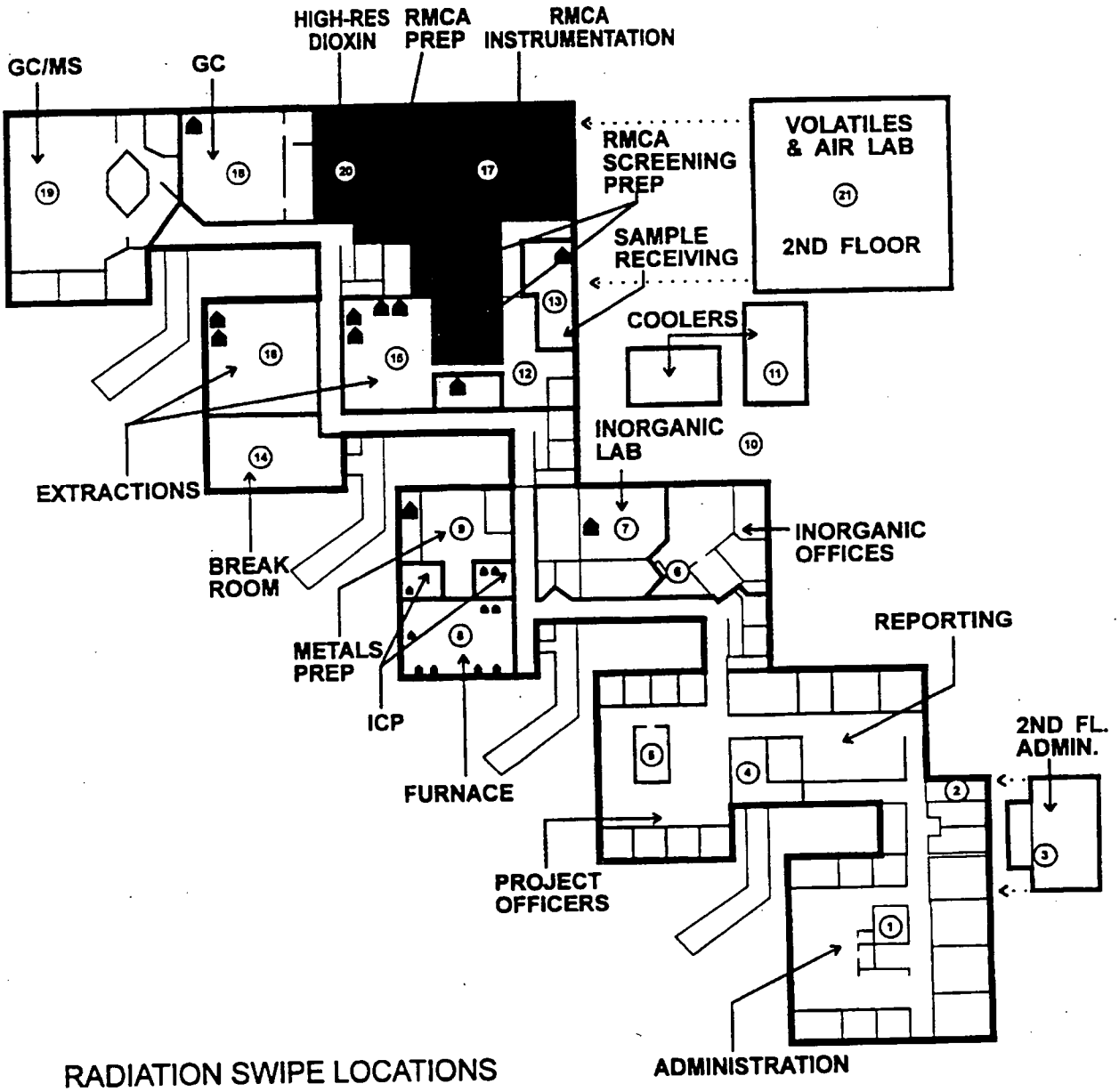
X. Film

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)



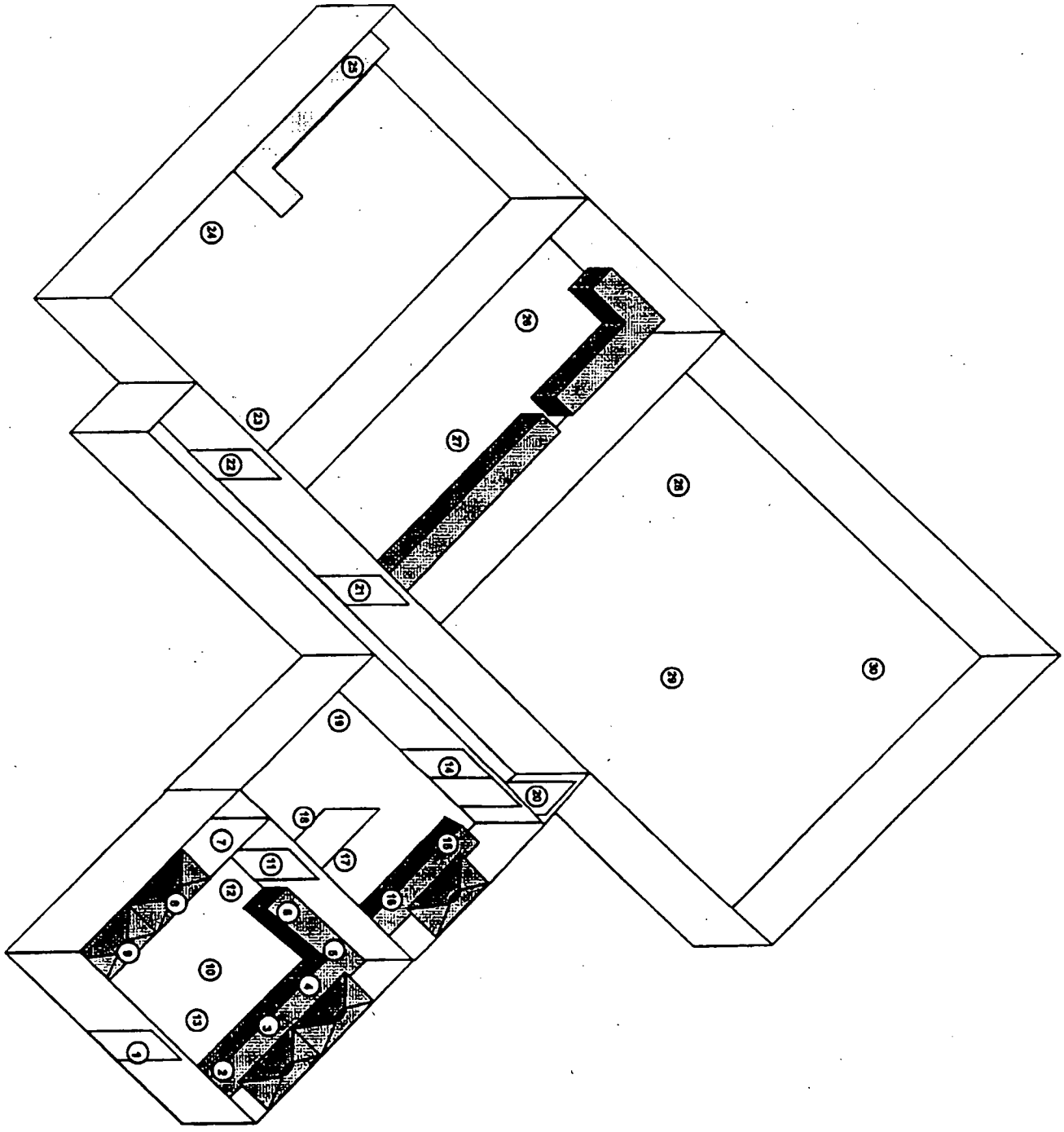
RADIATION SWIPE LOCATIONS

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

MICROSEEPS, INC.

Standard Operating Procedure for Glassware Washing

Controlled Copy No.

Mary Ellen Tenney
QA/QC Coordinator
Primary Author

Dominick Frollini Ph.D
Primary Reviewer

Carl Martin
Laboratory Director

Signature of Final Approval:

Patrick McLoughlin Ph.D.
Technical Director

Date: 8-25-99

1. Purpose and Applicability

The purpose of this standard operating procedure is to define the steps necessary to prevent contamination of analytical results that may result from the use of dirty glassware.

This procedure is applicable to all types of glassware used in the laboratory. Specific washing and rinsing steps particular to the type of glassware and the analytical method that will be used are defined in section 4 of this SOP.

2. Summary of Method

Cleaning of glassware methods should be considered both for substances to be removed and the analysis to be determined. Water-soluble items for general glassware uses are washed with hot soapy water and rinsed with tap water, then distilled water. Difficult residues may require more intense cleaning solutions such as detergents, organic solvents, acids, and dichromate cleaning solutions.

Analyst should dispose of materials properly and rinse glassware with tap water soon after use. Allowing residue to dry on glassware makes it more difficult to remove and may destroy the life of the vessels.

2. Responsibility

The analysts using the glassware are responsible for proper washing. Cleaning of glassware methods should be considered both for substances to be removed and the analysis to be determined. Water-soluble items for general glassware uses are washed with hot soapy water and rinsed with tap water and then de-ionized water. Difficult residues may require more intense cleaning solutions such as detergents, organic solvents, acids, and dichromate cleaning solutions.

3.1 It is the responsibility of the analyst to insure that all glassware being used is labeled.
3.2 Chemicals and samples shall be disposed of properly by the analyst, according to its waste type - hazardous or non-hazardous waste.

3.3 All persons using glassware shall rinse the vessels before placing them on the counter to be washed. Labels shall be read carefully, not only for safety considerations but also to make sure the glassware is being washed for its proper components.

4. Procedure

Specific cleaning techniques are outlined below for each type of glassware to be washed:

4.1 Ordinary Glassware

a. Wash with detergent (Alconox, Sparkleen or Citranox) and hot water
b. Rinse with tap water
c. Rinse with 1:1 HCl
d. Rinse with tap water
e. Rinse with de-ionized water three times

4.2 Evaporating Dishes

a. Scour out residue with a scrounge
b. Wash with (Alconox, Sparkleen or Citranox) and hot water
c. Rinse three times with tap water
d. Rinse three times with de-ionized water
e. Burn out in a 180°C oven
f. Cool in a desicator

4.3 Biochemical Oxygen Demand Bottles

a. Wash with detergent (Sparkleen Alconox, or Citranox) and hot water
b. Rinse once with tap water
c. Rinse with 1:1 HCl
d. Rinse with tap water three times
e. Rinse with de-ionized water three times
f. Burn out in a 180°C oven
g. Cool in a desicator

4.4 Chemical Oxygen Demand Flask

a. Rinse once with tap water
b. Wash with detergent (Sparkleen, Alconox, or Citranox) and hot water
c. Rinse with tap water three times
d. Rinse with de-ionized water three times
e.

4.5 Phenolics Flask

a. Wash with detergent Sparkleen, Alconox, or Citranox) and hot water
b. Rinse with tap water three times
c. Rinse with 1:1 HCl
d. Rinse with tap water.
e. Rinse with de-ionized water three times

4.6 Phosphate Flask

a. Rinse with tap water
b. Wash with detergent (Citranox)and hot water
c. Rinse with tap water three times
d. Rinse with 1:1 HCl
e. Rinse with de-ionized water three times

4.7 Surfactants Glassware

a. Rinse with tap water
b. Rinse with 1:1 HCl
c. Rinse with tap water three times
d. Rinse with deionized water three times

4.8 Metals Glassware

a. Wash with detergent (Citranox) and hot water
b. Rinse with tap water three times
c. Rinse with 1:1 HNO_3
d. Rinse with de-ionized water three times

4.9 Mercury Bottles

For bottles containing less than 0.01 ppm Hg:

a. Wash with detergent (Citranox) and hot water
b. Rinse with 1:1 NHO_3
c. Rinse with tap water
d. Rinse with de-ionized water three times

For bottles containing greater than 0.01 ppm Hg:

a. Soak in aqua regia for 1 hour
b. Rinse with tap water
c. Wash with detergent (Citrranox) and hot water
d. Rinse with 1:1 NHO_3
e. Rinse with tap water
f. Rinse with de-ionized water three times
g. Check the bottle for contamination prior to use for samples by using it to run an extra blank through the mercury procedure to ensure that it is non-contaminated.

4.10 Organic Glassware

a. Wash with detergent (Sparkleen, Alconox, or Citranox) and hot water
b. Rinse with tap water three times
c. Rinse with de-ionized water three times
e. Rinse with acetone three times

4.11 Oil and Grease Flask

a. Rinse with acetone
b. Wash with detergent (Sparkleen, Alconox, or Citranox) and hot water
h. Rinse with tap water three times
i. Rinse with de-ionized water three times
j. Rinse with acetone
f. Place in 105°C oven
g. Place in dessicator for storage

4.12 Cyanide Flask

a. Wash with detergent (Sparkleen, Alconox, or Citranox) and hot water
b. Rinse with tap water three times

c. Rinse with 1:1 HCl
d. Rinse with de-ionized water three times

4.13 Hard to clean residues and General Comments

4.13.1 More extensive cleaning procedures for hard-to-clean residues and general comments for cleaning are as follows:

4.13.2 Volumetric glassware can be thoroughly cleaned by mixing 4 grams (g) of sodium hydroxide, 4 g of sodium hexametaphosphate, and 8 g of trisodium phosphate into a liter of water.

4.13.3 Chromic acid solution is a powerful cleaning agent; however, it is extremely dangerous and should be handled with care. Chromic acid is made by adding 1 liter of concentrated sulfuric acid to 35 milliliters (ml) of saturated sodium dichromate. This solution is placed into the vessel to be cleaned for approximately 15 to 30 minutes and then poured back into the storage bottle. After treatment with chromic acid, the glassware is then rinsed with tap water and then distilled water.

4.13.4 Persistent greasy layers or spots may be removed with acetone or with a warm solution of sodium hydroxide. Removal of oil and grease from glassware can be achieved by adding 1 g of sodium hydroxide into 50 ml of warm water and allowing the solution to stand for about 15 minutes to react. Rinse glassware with water followed by diluted hydrochloric acid. Rinsing usually removes stubborn spots. Alcoholic potassium hydroxide is also effective in removing grease.

4.13.5 Burets can be washed with detergents and rinsed with distilled water. Under no circumstances should burets be dried; they should only be conditioned with the titrant before use. As a general rule, solutions should not be stored in burets.

4.13.6 Spectrophotometric cells should be kept free of fingerprints, smudges, and film residues. Detergents can be used for cleaning but do not allow soaking for long periods of time. Absorption cells can be rinsed with either nitric acid or organic

solvents prior to use. Rinsing with acetone or alcohol for storage is preferred. Scratched cells should be replaced and taken from service.

4.13.7 Glassware used for phosphorus analysis should never be washed with detergents containing phosphate additives, whereas glassware used for ammonia or total kjeldahl nitrogen should be rinsed with ammonia-free water.

4.13.8 Glassware used for the determination of organics can be cleaned by washing with chromic acid for 15 minutes and rinsing with tap water and then distilled water. The glassware then can be dried by either an acetone rinse for immediate use or in an oven prior to storage.

4.13.9 When hazardous or heavily contaminated samples are encountered, disposable glassware may be preferred.

4.13.10 Following any of the washing procedure described in this section, the glassware should then be washed according to the appropriate method described in section 4.

5. Quality Control

Volumetric glassware is glassware that has been accurately calibrated for precise measurement of volume. The accuracy by which volumes of solutions are measured depends on the precision of the volumetric glassware. Volumetric glassware is calibrated "to contain" (TC) or "to deliver" (TD) a definite volume of liquid. TD volumetric pipets are routinely used in the laboratory. Volumetric pipet (TC) tips are held against the wall of the receiving vessel until flow has stopped. Do not ever blow out the remaining liquid left in the tip of the pipet. All apparatus used for delivering liquids should be volumetrically clean(no beading of liquids on the apparatus walls), this will ensure accurate volume dispensing.

Analysts should dispose of materials properly and rinse the glassware with tap water soon after use. Allowing residue to dry on glassware makes it more difficult ot remove and may destroy the life of the vessel.

Final disposal of disposable containers, wastes, spent chemicals and chemical containing wastes are handled in accordance with the Microseeps Chemical Hygiene Plan and the Microseeps Waste Disposal Plan.

6. Safety

Safety is a major concern throughout the laboratory environment, not only at the analytical bench, but also for the consideration of the dishwasher.

Anyone providing analytical cleaning support shall wear a lab coat, safety glasses, and protective gloves before handling any glassware.

7. Equipment and Reagents

7.1 Equipment
- 7.1.1 Sinks
- 7.1.2 Scrub brushes
- 7.1.3 Drying rack

7.2 Reagents
- 7.2.1 Tap water
- 7.2.2 Detergents
- 7.2.3 Deionized water
- 7.2.4 Nitric acid (1:1)
- 7.2.5 Aqua Regia (25% by volume conc. HNO_3 in concentrated HCl). To prepare Aqua Regia: mix 3 parts of concentrated HCL and 1 part of concentrated HNO_3. Prepare just before use.
- 7.2.6 Acetone
- 7.2.7 Dichromate solution (chromic acid)
- 7.2.8 Hydroxide solution

8. Documentation and References

Not Applicable

Microseeps, Incorporated

Analytical Method AM20GAx Standard Operating Procedure for the Analysis of Biodegradation Indicator Gases

Controlled Copy No. ____

Controlled Document
Confidential

Dominic Nestasie
Laboratory Director

Signature of Final Approval:

Patrick McLoughlin, Ph.D.
Author and Technical Director

3-17-03
Date

1.0 Scope and Application

Method AM20GAx is used to determine the concentration of biodegradation indicator gases in vapor samples. Specifically, Method AM20GAx is used to determine the dissolved concentration of the following gases:

Gases	CAS Number
Carbon dioxide	124-38-9
Oxygen	7782-44-7
Nitrogen	7727-37-9
Hydrogen	1333-74-0
Methane	74-82-8
Ethane	74-84-0
Ethene	74-85-1
Propane	74-98-6
Propene	115-07-1
n-Butane	106-97-8
i-Butane	75-28-5
Carbon Monoxide	630-08-0

This method is recommended for use by, or under the supervision of, analysts experienced in sample preparation, the operation of gas chromatographs and in the interpretation of chromatograms.

2.0 Method Summary

The sample gas is analyzed with a gas chromatograph capable of simultaneous analysis of all of the target analytes from a single 10 mL gas sample. A single injection of gas from integral, simultaneously filled sample loops is used to assure consistent injection volume. The permanent gases are analyzed using a thermal conductivity detector (TCD). The light hydrocarbons are analyzed using a flame ionization detector (FID). Hydrogen is analyzed using a reduction gas detector (RGD). The data are transferred to a microcomputer, converted to digital format, stored, and processed using a chromatography data system.

2.1 Definitions

Instrument Flush: The front end of the sample loop is flushed with ultra high purity helium injected into the loop directly from the cylinder to remove possible interference by ambient air and to avoid cross contamination between samples.

Method Blank: A sample analyzed by all three detectors that consists of ultra high purity helium. The method blank is free from the analytes of interest

Laboratory Control Sample: A sample spiked with verified known amounts of analytes. A LCS is used to assess the performance of the measurement system.

Matrix Spike and Matrix Spike Duplicate: A sample prepared by adding a known concentration of target analyte to a specific amount of sample. Matrix spikes are used to determine the effect of sample matrix on a method's recovery efficiency.

3.0 Apparatus and Materials and Operating Conditions

3.1 Apparatus

Gas Chromatograph: The chromatograph designed and built by Microseeps is equipped with multiple packed columns and multi-port valves, a TCD, a FID, a RGD, and multiple sample loops. The FID, which is also built by Microseeps, is of a special design that allows considerably more sensitivity than commercially available models. To increase the working range of the system, there are two outputs to the FID. Thus, it is a four-channel system: (1) FID low; (2) TCD; (3) RGD; and (4) FID high. As discussed in Section 5.3, each channel is calibrated separately. This instrument provides rapid turn-around for consecutive analyses and a clean baseline for accurate, reproducible results.

3.2 Materials

- Sample vials (Supelco, Inc, Bellefonte, PA or equivalent)
- Syringe: locking gas tight

3.3 Operating Conditions

Gas Chromatograph:

• Sample Loop Temperature:	ambient
• Thermal Conductivity Detector Temperature:	100°C
• Flame Ionization Detector Temperature:	ambient
• Reduction Gas Detector Temperature:	280°C
• Oven Temp.:	100 °C. isothermal
• TCD Signal Range:	1
• FID Signal Range:	variable depending on concentrations
• RGD Signal Range:	1
• He Carrier Gas Regulator Pressure:	60 psig
• Sample carrier flow:	30 mL/min.
• Reference flow:	30 mL/min.
• N2 Carrier Gas Regulator Pressure:	25 psig

- Sample carrier flow: 25 mL/min
- Valve Air Pressure: 60 psig.

3.3.1 Interferences

The most likely source of "interference" is ambient air. Due to the relatively high concentrations of oxygen and nitrogen, a very small amount of air as a contaminant will dramatically affect the results. The analyst must take great care to ensure that air is flushed from the gas tight syringe before sample preparation and that no air has entered the syringe or needle prior to injection of the sample into the gas chromatograph.

Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. An unrestricted flow (Instrument flush) of pure carrier gas from a 10 psig source should be allowed to flow through each sample loop for 30 seconds prior to each analysis.

As required, the analyst should demonstrate the absence of carryover contamination by analysis of the contents of the sample loop when purged with carrier gas. This demonstration should be performed when carryover contamination is suspected (after high samples). In the event that 'ghost peaks' (peaks similar to previous sample) appear when a pure carrier gas sample is analyzed (method blank), measures should be taken to eliminate the carryover contamination.

4.0 Reagents

- Helium (UHP Gas)
- Nitrogen (UHP Gas)
- Certified Commercial Gas Standards

4.1 Standard Preparation Procedures

Calibration standards are prepared by using the procedures below:

4.1.1 Vial Preparation

Headspace vials used for instrument calibration standards for this method are prepared as follows:

- Crimp and cap each vial, with stopper septa.
- Evacuate each vial to vacuum below 100 millitorr.
- Flush each vial to atmospheric pressure with the vial preparation gas. The gas used depends upon the detector that is being calibrated and is specified in Table 4.1 below:

Table 4-1

Detector	Vial Preparation Gas	Standard Mix Vendor
FID	Nitrogen	Spectra
TCD	Helium	Scotty
RGD	Nitrogen	Spectra and Scotty

4.1.1 Preparing Calibration Standards

Instrumentation is calibrated using dilutions of custom certified gas mixes. (Refer to Table 4-2 for the correct amounts of standard mix and vial preparation gas to inject into prepared vials.)

- Prepare the correct number of vials as specified above for the detector being calibrated.
- Inject the specified amount of standard by extracting it from the standard mix gas cylinder using a gas-tight syringe and injecting it into a prepared vial.
- Then the specified amount of vial preparation gas is added to the same vial.

The dilution factor of one is achieved by directly injecting the standard gas mix from the cylinder into the GC.

Table 4-2

Dilution Factor	FID		TCD		RGD	
	Standard Gas Mix	Prep. Gas	Standard Gas Mix	Prep. Gas	Standard Gas Mix	Prep. Gas
1	Direct inject mix into GC		Direct inject mix into GC		Direct inject Spectra mix into GC	
2	21cc	0 cc	21cc	0cc	21cc Scotty	0cc
3	15cc	9cc	N/A	N/A	N/A	N/A
10	4cc	15cc	4cc	15cc	4cc Spectra	15cc
100	0.5cc	28.5cc	0.5cc	28.5cc	0.5cc Spectra	28.5cc

4.2 Quality Control Sample Preparation

Quality control samples are prepared as indicated below.

4.2.1 Laboratory Control Sample (LCS) and LCS Duplicate (LCSD)

The LCS and LCSD are directly injected into the sample port from a second source certified standard gas mix. A needle is plumbed to the cylinder and used to inject the gas into the sample loop.

4.2.2 Matrix Spike (MS) and Matrix Spike Duplicate (MSD)

MS and MSDs are prepared when clients' request and send sufficient numbers of aliquots to prepare them (e.g. one 40 ml vial each for the MS and the MSD). They are prepared, one at a time, as follows:

- Using a clean 50ml gas-tight locking syringe, withdraw a volume of water from the bottom of the sample vial.
- Withdraw 10 cc of the certified standard gas used for preparing the LCSs and lock the syringe.
- Shake the syringe by hand (for use a wrist action shaker) for five minutes.
- The equilibrated MS and/or MSD is/are now ready to be analyzed.

4.2.3 Method Blank

Method blanks are made up of ultra high purity helium injected into a vial and then into the instrument.

4.3 Glassware and Storage Requirements for Reagents and Standards

Reagents are stored at ambient room temperature and all standards are prepared shortly before analyses.

5.0 Procedure

The sample must be received and processed via Microseeps SOP for those procedures. Gas samples are shipped and received at a positive pressure, which eliminates a cross-contamination issue during sample shipment. They are stored at ambient temperature to avoid creating a pressure reduction in the samples upon cooling. Such a pressure reduction could lead to a drawing of atmospheric gas into the sample. A sample batch consists of twenty or fewer samples run during an eight-hour work shift.

Analysts who use this method have been certified for the method by running Initial Demonstration of Proficiency Samples. The IDOPs are an identical set of 4 samples (PM01/AM20Gax uses tap water and AM20Gax uses known standard concentrations) run

consecutively. IDOPs are run any time there is significant change to an instrument, method, or in the Training procedure for training a new analyst.

The percent relative standard deviation (%RSD) is calculated for the IDOPs by:

$$\%RSD = \frac{\sigma_{n-1}}{S} \times 100\%$$

Where:

% RSD = the percent relative standard deviation.
σ_{n-1} = the standard deviation about the mean.
S = the mean response $(X_1 + X_2 + X_3 + X_4)/4$

The %RSD must be less than 20%. If this criterion is not met, the system is checked and the IDOP samples are re-analyzed.

5.1 Sample Preparation

Samples that are collected using the Bubble Strip Sampling Technique, Microseeps Sampling Method SM9, do not require additional preparation prior to analysis.

Samples that are collected as waters and are to be analyzed for dissolved gases (CO_2, N_2, O_2) must be prepared using Microseeps Standard Operating Procedure PM01G.

Samples that are collected as gases, for example from a soil gas survey or from the headspace of a microcosm sample, need not be collected by a Microseeps sampling method, nor do they require additional preparation.

5.2 Analysis

5.2.1 If the sample is prepared via DGPM 01, it can be injected from the gastight syringe in which it is prepared by inserting the needle of the syringe through the septum on the "sample in" port. If the sample is a calibration standard, a bubble strip sample (SM9), or a gas, the septum inlet to the "sample in" port of the GC must be removed and a luer-lock needle receptacle is plumbed to the "sample in" port in place of the needle. A needle is attached to the luer-lock receptacle and inserted through the septa of the calibration standard, bubble stripped sample, or gas sample.

In order to initiate analysis and introduce the sample into the GC sample loop, a needle is attached to the entry port on the GC and inserted through the sample septum. The flow through the sample loop is monitored by a flow meter connected to the sample-loop vent-port on the gas

chromatograph. If the loop is not properly pressurized, this is reflected on the flow meter immediately.

5.2.2 If the flow is insufficient the vial is rapidly removed. A clean, gas-tight syringe is flushed with He, the plunger of that syringe is completely depressed, and the needle of that syringe is inserted through the sample septum. The plunger of that syringe is allowed to expand completely. The syringe is removed from the sample and its contents are discharged into an appropriate receptacle. 22 ml. of nitrogen are then used to pressurize the sample vial, the dilution is noted, and the sample is injected onto the GC as though it were a properly over-pressurized sample.

5.2.3 Once the flow out of the sample loop ceases (3 seconds if DGPM 01 is used) the sample loop valves are activated.

5.2.4 Once the sample loop valves are activated, the ports to and from the sample loop are flushed with ultra high purity helium injected into the loop directly from the cylinder to remove any interference from ambient air and to avoid cross contamination between samples.

5.3 Calibration and Results

5.3.1 The standard calibration gas should be introduced in the same manner as described in section 5.2.1 above. Measured peak areas are converted to concentrations using certified commercial gas standards. Dilutions are made to achieve multi-point calibration curves for each detector.

5.3.2 Initial calibration is accomplished by analyzing multiple standards of appropriate calibration ranges. Table 5.1 specifies the approximate concentrations of calibration standards for each detector.

Note: Due to the nature of preparing custom gas standards, the component concentration can fluctuate between purchased lots. This is accounted for during method/calibration development. These results will be used to establish a multi-point calibration curve.

Acceptance Criteria: A linear fit to an area response versus concentration plot is formed with the origin forced to zero, and the calibration is accepted for use if r^2, the coefficient of determination is ≥ 0.995.

Corrective Action: If the acceptance criteria specified above is not met, the reason is determined and a new set of calibration standards are analyzed.

5.3.3 An Initial Calibration Verification (ICV) standard immediately follows the initial calibration. The ICV is made up separately from the same stock standards used to make up the ICAL. (Second source requirement in LCS) Acceptance criterion for the ICV is an instrument response of $\leq$ (less than or equal to) 20% (%D).

Acceptance Criteria and Corrective Action: If the instrument response for the ICV standard varies by more than 20% (%D), the analyst will not analyze samples until either the reason is determined and the problem is corrected, or a new multi-point calibration is analyze

Table 5.1

COMPOUND	FID-LOW (ppmv)	FID-HIGH (ppmv)	TCD (% V)	RGD (ppmv)
carbon dioxide	-	-	15, 7.5, 0.15	-
oxygen	-	-	7, 3.5, 0.07	-
nitrogen	-	-	66.5, 33.3, 0.665	-
hydrogen	-	-	-	100, 25, 2.5, 0.25
methane	300, 100, 30, 3	1000, 300, 100, 30	4.5, 2.25, 0.045	-
ethane	100, 33.3, 10, 1	1000, 100, 33.3, 10	-	-
ethene				
propane	100, 33, 10, 1	100, 33.3, 10	-	-
propene				
i-butane				
n-butane				

5.3.4 An initial calibration blank follows the ICV. The blank is made up of the carrier gas. Compounds must not be detected above the reporting limits.

Corrective Action: If the blank does not meet the acceptance criterion, another blank is injected until the results are within the acceptance criterion.

5.3.5 The analytes of this method are indicators. Every attempt to achieve and deliver precise results is made. However, it is realized that for indicator parameters measuring the range of the analyte concentration (*i.e.* is the concentration of methane gas >1 mg/l or < 0.1 mg/l?) is the primary goal of employing these analyses. The calibration range is chosen to extend over the entire useful bio-indicator concentration range. If the concentration of an analyte exceeds that of the highest calibration standard, the concentration is calculated by assuming detector response linearity and using an extrapolation of the calibration plot.

5.3.6 The unique combination of analytes used in this method makes it impractical to find a second source for the calibration mix. To insure that the calibration is accurate, the first use of a new standard is as a CCV against a calibration prepared with the old standard. If this CCV passes the standard CCV criteria, the new standard is placed into routine use. This insures that as the original source of the master mix from which our standards are prepared is switched,

Microseeps checks the reported calibration of the new against the old. If the client requests a formalized check of the standard, a calibration check standard can be analyzed and included in the clients report provided that the request for this service is made prior to collection of the samples.

5.4 Quality Control

The following quality control samples shall be analyzed with each analytical batch of twenty or fewer samples.

5.4.1 A Continuing Calibration Verification: The CCV is made up separately from the same stock standards used to make up the initial calibration. The acceptance criterion for the CCV is a percent drift of ± 20%.

Corrective Action: If the CCV fails, the instrument shall be recalibrated, and all samples since the last acceptable calibration shall be reanalyzed, provided sufficient sample volume is present and the samples have not been compromised by exposure to air.

5.4.2 A Continuing Calibration Blank: A CCB follows each CCV. The blanks are made up of the carrier gas. The acceptance criterion for the blank is the result must be less than the reporting limits for all compounds.

Corrective Action: If the blank does not meet the acceptance criterion, another blank is injected until the results are within the acceptance criterion.

5.4.3 Laboratory Control Sample and Laboratory Control Sample Duplicate: The LCS and LCSD are prepared at a mid-calibration range and comes from a source other than the calibration standards. The acceptance criterion is a percent recovery between 75% and 125% and a Relative Percent Difference of less than or equal to 20%.

$$Percent\,Recovery = \frac{Measured\,Value}{True\,Value} \times 100\%$$

$$RPD = \frac{|C1 - C2|}{\frac{C1 + C2}{2}} \times 100\%$$

Where: C1=LCS
C2=LCSD

Corrective Action: If the LCS fails, a new LCS is prepared and analyzed. If the new LCS falls within the acceptance criterion, analysis continues. If the new LCS fails, analysis is stopped and the instrument is checked with a series of standards to determine the cause. Once the cause is determined and the instrument repaired, calibration is conducted and analysis continues.

5.4.4 Matrix Spike and Matrix Spike Duplicate: Matrix spikes and spike duplicates are analyzed for water samples only when requested by a client and sufficient sample aliquots are provided. Acceptance criterion is a percent recovery between 70% and 130%, and a relative percent difference of less than or equal to 20%.

Corrective Action: If the matrix spike and spike duplicate fails but all the other quality control samples are within the acceptance criteria, a matrix interference is noted in the Case Narrative.

5.4.5 Method Blank: A method blank is analyzed with each sample batch. The blanks are made up of UHP helium. The acceptance criterion for the blank is the result must be less than the reporting limits for all compounds.

Corrective Action: If the blank does not meet the acceptance criterion, another blank is injected until the results are within the acceptance criterion.

5.4.6 Contingency for Handling Out of Control or Unacceptable Data

If the requirements set forth in section 5.4 are not met, the analytical program will be terminated until the cause is determined and a solution is affected. All samples associated with out of control quality control samples (with the exception of matrix interference) must be reanalyzed provided another vial of sample has been provided by the client. If quality control acceptance criteria cannot be met using the corrective action above, a detailed check of the analytical system is made. Reagents, standards, and other quality control samples are re-prepared and analyzed. If problems persist, sample analysis will be halted and the Technical Director shall be contacted immediately to determine the cause and implement corrective action.

Any data submitted with unacceptable quality control sample results shall be qualified in a case narrative. The narrative should indicate the out of control event that occurred, the corrective action that was taken, and any other pertinent information to inform the client of exactly what occurred.

5.4.7 An experienced analyst shall examine all chromatograms.

5.4.8 Through out analysis the gas samples are injected mechanically into the GC flow path utilizing a sample loop to achieve a uniform sample size from a flow directly from the sample preparation syringe. The uniform sample size achieved using the sample loop assures consistent and accurate results. Table 5.2 (see next page) gives example data from a study performed via this analysis. That data can also be used for accuracy and precision estimates.

Table 5.2
Example Data for Precision and Accuracy Studies

	Carbon Dioxide	Oxygen	Nitrogen	Methane	Hydrogen	Methane	Ethane	Ethylene	Propane	Propylene	Iso-Butane	N-Butane
REP. #	(%v)	(%v)	(%v)	(%v)	(PPMV)	(PPMV)	(PPMV)	(PPMV)	(PPMV)	(PPMV)	(PPMV)	(PPMV)
1	0.1221	0.0670	0.5744	0.0410	0.1118	0.2512	0.0525	0.0453	0.0461	0.0581	0.0473	0.0358
2	0.1267	0.0690	0.6020	0.0428	0.1122	0.2608	0.0518	0.0468	0.0521	0.0465	0.0439	0.0407
3	0.1207	0.0657	0.5838	0.0446	0.1247	0.2812	0.0509	0.0485	0.0529	0.0588	0.0436	0.0405
4	0.1193	0.0667	0.6036	0.0444	0.1244	0.2779	0.0549	0.0460	0.0461	0.0536	0.0549	0.0476
5	0.1261	0.0703	0.5860	0.0439	0.1120	0.2894	0.0551	0.0497	0.0520	0.0549	0.0417	0.0460
6	0.1193	0.0665	0.5861	0.0478	0.0943	0.2970	0.0515	0.0467	0.0458	0.0542	0.0435	0.0514
7	0.1227	0.0732	0.5748	0.0353	0.1296	0.3053	0.0532	0.0473	0.0485	0.0584	0.0483	0.0535
AVERAGE	0.1224	0.0683	0.5872	0.0428	0.1156	0.2804	0.0528	0.0472	0.0491	0.0549	0.0462	0.0451
KNOWN	0.1500	0.0700	0.6649	0.0450	0.0999	0.1500	0.0500	0.0500	0.0500	0.0500	0.0500	0.0500
STD. DEV.	0.003	0.003	0.012	0.004	0.012	0.019	0.002	0.001	0.003	0.004	0.004	0.006

5.4.9 The gas matrix for this analysis minimizes the opportunity for matrix effects. If the gas is prepared from a matrix other than that which is injected into the GC (*e.g.* prepared through headspace extraction via Microseeps SOP-PM01), the client should request that matrix spike (MS) and matrix spike duplicate (MSD) analyses be conducted and should supply sufficient sample volume. Since matrix effects are extremely site dependent, the MS and MSD are not part of the regular analytical quality assurance program.

In order to determine analytical precision, relative percent difference (RPD) is calculated between the matrix spike and matrix spike duplicate results using the following formula:

$$\%RPD = \frac{|C_1 - C_2|}{\left|\frac{C_1 + C_2}{2}\right|} x100$$

Where: C_1= Matrix Spike Result
C_2= Matrix Spike Duplicate Result

In order to determine accuracy, percent recovery of the matrix spike is calculated using the following formula:

$$\% \text{ Recovery} = \frac{\text{concentration found}}{\text{concentration added}} \text{ X } 100$$

5.4.10 All of the target analytes are gases at room temperature so the opportunity for carry over is negligible. Because of the configuration of the GC used in this analysis, any possible carryover would only manifest itself as a ghost peak, well out of the RT windows of any analytes and thus never misinterpreted. For these reasons, samples that have high concentrations of analytes do not need to be followed by a blank analysis.

5.5 Capturing and Submitting Data

5.5.1 The output of the chromatograph is directed to a microcomputer where the signal is converted to digital format, stored, and processed using a chromatography data system.

5.5.2 Automated valve control: Digital control is provided by the microcomputer though the chromatography data-system software. This control provides constant start and stop times for directing carrier gas flow. The event times are programmed and saved using the method editor module of the software.

5.5.3 Calibration records are generated in electronic and hard copy formats and stored. All such records will be maintained in the laboratory during the course of the project and thereafter as determined by the client.

5.5.4 The instrument produces four channels of data, and an output is generated from each sample for each channel. Each channel is used for a distinct set of analytes, in a distinct range. The channel designations are listed in Table 5.3 below, as are the analytes and range specification for each analyte.

Table 5.3

FID	FID/10	TCD	RGD *
iso-butane (low)	iso-butane (high)	carbon dioxide	hydrogen
n-butane (low)	n-butane (high)	nitrogen	
propene (low)	propene (high)	oxygen	
propane (low)	propane (high)	methane (high)	
ethane (low)	ethane (high)	carbon monoxide	
ethene (low)	ethene (high)		
methane (low)	methane (high)		

* If hydrogen is not a required analyte, the GC may be run without this channel activated. This reduces the generation of unused records and the consumption of power.

If an analyte is not detected on the most sensitive channel, it is recorded as a non-detect. That channel, designated low, is used until the detector output as read from the chromatograph of the channel, exceeds 2V. At that point the next most sensitive detector ("mid" if available, or "high" otherwise) is used. If that saturates, the "high" detector is used if available. Otherwise, the sample is diluted and re-analyzed. The saturation limit, in terms of concentration, varies machine to machine and calibration-to-calibration, so universal cut-offs cannot be supplied here. The final choice of which channel to use when reporting results is up to the experienced analyst and depends upon the sample response, the calibration, and the history of the instrument.

5.5.5 This analysis produces concentration of the analyzed gas in PPMV or % V. If the sample was collected via the bubble-strip method (Microseeps SOP SM9) or prepared through static headspace preparation (Microseeps SOP DGPM 01), the gas phase concentrations can be used to specify sample water concentrations via the calculations presented in those Standard Operating Procedures.

6.0 Secondary Data Review

All analytical data must undergo a minimum of a two-tiered review. The analyst first reviews the data for completeness and accuracy. The data is then submitted to the Group Lead Analyst for final review and the data is entered into the LIMS. Once approved at this level, the data is released as a final report.

7.0 Reporting Limits

The reporting limits for this analysis are listed in table 7.1 below. The method detection limits for this procedure must be determined by conducting annual MDL Studies. Those MDLs must be less than the reporting limits specified below. MDL studies are also performed when there is reason to suspect that method sensitivity has changed. MDLs are determined by analyzing seven aliquots of a known standard (estimated to be 3 to 5 times the lowest concentration that can be seen above noise level) through the procedure, and calculating the mean and standard deviation. Once the seven aliquots are run the results are given to the Quality Systems Coordinator. The MDL studies are kept on file in the Quality Systems Office.

Reporting Limits
Table 7.1

Parameter	Reporting Limit	Units
Carbon Dioxide	0.02	%V
Oxygen	0.02	%V
Nitrogen	0.04	%V
Hydrogen	0.02	ppmv
Methane	0.06	ppmv
Ethane	0.01	ppmv
Ethene	0.01	ppmv
Propane	0.01	ppmv
Propene	0.01	ppmv
n-butane	0.01	ppmv
i-butane	0.02	ppmv

7.2 Conversion Factors

This procedure is used to measure the volume concentration of the analytes in a gas. Two methods are used to extract that gas from the groundwater. The conversion factors that are used to convert the concentration of the analytes in the water from the concentration of the analytes as they are measured using this method, are specific to the collection or preparation method and can be found in either SOP-SM9 or SOP-DGPM 01.

8.0 Safety

Gloves, proper eye protection, and a laboratory coat shall be worn when handling samples and standards. The major hazard in this laboratory area is stick from needles. All needles must be capped when not in use and when moving about the laboratory. The proper way of capping a needle is to place the cap on the laboratory bench and direct the needle into the cap. A needle is never to be directed into a cap while the cap is being held.

All compressed gases are to be moved using a dolly made for transporting gases and shall be chained in place when in the laboratory. The chain shall be tightened sufficiently to keep the cylinder upright if jostled.

9.0 Laboratory Waste

Samples are kept for 30 days following analysis. There is no hazardous waste generated during this analytical process. The analysts dispose of the sample vials in the general laboratory trash according to Microseeps Standard Operation Procedure for Waste Disposal (SOP-ADM 14).

9.1 Waste Minimization

Where possible, Microseeps takes steps to minimize the amount of waste generated in the laboratory by using substitution, where possible, and good chemical handling procedures. For specific information on waste minimization consult SOP-ADM 14.

10.0 References

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste. SW-846, 3rd ed., Office of Solid Waste and Emergency Response, Washington, DC. 1986.

Newel, B.S., RSKSOP-175, Sample Preparation and Calculations for Dissolved Gas Analysis in Water Samples using a GC Headspace Equilibration Technique. Revision No. 0, August 1994.

Newel, B.S., RSK-SOP-147, Gas Chromatographic Analysis of Gaseous Samples for Part-Per-Million Levels of Nitrous Oxide, Methane, Ethylene, and Ethane. Revision No. 0, August 1993.

American Society for Testing and Materials, Standard Practice for Analysis of Reformed Gas by Gas Chromatography. Annual Book of ASTM Standards. Vol. 14.02, 1994.

Kampbell, D.H. and Vandegrift, S.A., Analysis of Dissolved Methane, Ethane, and Ethylene in Ground Water by a Standard Gas Chromatographic Technique. Journal of Chromatographic Science. Vol. 36, May 1998.

Controlled Document
Contents

Microseeps, Inc.

Standard Operating Procedure for the Preparation of Samples for the Analysis of Biodegradation Indicator Gases

Controlled Copy

Controlled Copy No. _____

Dominic Nestasie
Laboratory Director
Primary Reviewer

Signature of final Approval:

Dr. Patrick McLoughlin
Technical Director and Author

2-6-03
Date

1.0 Purpose and Application

Method PM01G is used to prepare a chemically equilibrated gas-phase (*i.e.* vapor) headspace above a fixed volume of aqueous sample. The composition of that headspace can then be analyzed by Microseeps' AM20GAX and the results can be used to calculate the concentration of gases in the sample water. Conceptually, this method can be used for any substance that can be detected in the gas phase, but practical considerations of sensitivity, vapor pressure and air/water partitioning limit the technique. Method DGPM 01 is used for the following gases:

Gases	CAS Number
Carbon dioxide	124-38-9
Oxygen	7782-44-7
Nitrogen	7727-37-9
Methane	74-82-8
Ethane	74-84-0
Ethene	74-85-1
Propane	74-98-6
Propene	115-07-1
n-Butane	106-97-8
i-Butane	75-28-5

This method is recommended for use by, or under the supervision of, analysts experienced in sample preparation, the operation of gas chromatographs, and in the interpretation of chromatograms.

2.0 Method Summary

A water sample is transferred from a 40 mL vial into a gas-tight syringe and an analyte-free; inert gas is used to create headspace above that water. At equilibrium, the headspace should contain each of the analytes in proportion to the air/water-partitioning coefficient of the analyte. Agitation of the syringe accelerates the onset of equilibrium. Following that agitation, the equilibrated sample is ready for analysis (*e.g.* by method AM20GAX) or storage.

3.0 Apparatus, Materials and Operating Conditions

3.1 Apparatus

- Sample vials (Supelco, Inc, Bellefonte, PA or equivalent).
- Syringe: locking gas tight
- Luer lock valve

3.2 Materials

- Ultra-pure Helium supply.

3.3 Operating Conditions

The following situations are cases where sample "interference" could occur.

3.3.1 Ambient Air

The most likely source of "interference" is ambient air. Due to the relatively high concentrations of oxygen and nitrogen, a very small amount of air as a contaminant will seriously affect the results. The analyst must take great care to ensure that air is flushed from the gas tight syringe before sample preparation and that no air has entered the syringe or needle prior to injection of the sample into the gas chromatograph.

3.3.2 Equipment not supplied by Microseeps

Upon occasion a sample will be collected in a vial that was not supplied by Microseeps. In such cases, the needle on the gas-tight syringe sometimes gouges the septum of the sample vial, rather then simply tearing it. This gouged out portion may occlude the needle and frustrate further use of it. As such, the gas-tight syringe should be fit with the water collection needle before the plunger is inserted into it and depressed.

3.3.3 Cross Contamination

The gases that are the analytes are extremely volatile. Carryover is generally not a problem. However, agents, such as surfactants, may be present in some samples and those agents can alter the partitioning coefficient and even present some chance of carryover. All syringes and re-useable needles should be cleaned thoroughly between sample preparations.

4.0 Reagents

Not Applicable

5.0 Procedure

The sample must be received and processed via Microseeps SOP for those procedures. Samples are stored in a refrigerator at 4 °C and allowed to equilibrate to room temperature prior to sample preparation.

5.1 Sample Preparation Steps.

1. Using a clean 50ml gas-tight locking syringe, withdraw a volume of water from the bottom of the sample vial.

2. Withdraw a volume of an inert gas from a reservoir and lock the syringe.

3. Shake the syringe by hand (or use a wrist action shaker) for five minutes.

4. The equilibrated sample is now ready to be analyzed or injected into a vial for storage for analysis at a later date.

5.2 Calibration and Results

Not Applicable.

5.3 Sample Analysis

Not Applicable.

5.4 Quality Control

Most of the quality control sample analyses that are "typical" of EPA methods, are not applicable to this analytical method due to the target analytes being naturally occurring substances in water. The most likely result of any analytical error is compromising the sample through exposure to ambient air. Since two of the analytes (nitrogen and oxygen) make up nearly 80% and 20% respectively, of the air all around us, it is virtually impossible to generate a truly inert "prep-blank". The best way to detect procedural error is through careful review of all analytical results, insuring that the oxygen and nitrogen concentration, and their ratio, truly reflects groundwater geochemistry. The concentration of nitrogen is typically between 10 and 20 mg/l, but the oxygen concentration can be much lower. In anoxic waters the nitrogen/oxygen ratio is typically above five. Ratios below this suggest significant sample contamination. A reanalysis should be performed to insure that the source of the contamination was not simple unsystematic error in the preparation.

5.4.1 Matrix Spikes/Matrix Spike Duplicates

To insure that there are no matrix effects in a client's samples and to produce client specific accuracy and precision data, clients should request matrix spike (MS) and matrix spike duplicate (MSD) analyses during the bottle order process, collect those samples while on site, and submit them as separate samples on the chain of custody to the laboratory. These analyses involve the analysis of an analyte-fortified client sample. The recovery efficiency of that fortification can reveal matrix effects, and the reproducibility of the result of the analysis of the fortified sample reveals client sample-specific precision information. Since matrix effects are extremely site dependent, the MS and MSD are not part of the batch analytical quality assurance program.

5.5 Capturing and Submitting Data

Not Applicable.

6.0 Secondary Data Review

Not Applicable.

7.0 Reporting Limits

Reporting limits for this method are specified below in Table 7.1.

Table 7.0
Reporting Limits

Analyte	Reporting Limit	Water Units
Carbon dioxide	0.6	mg/L
Oxygen	0.15	mg/L
Nitrogen	0.4	mg/L
Methane	0.015	µg/L
Ethane	5	ng/L
Ethene	5	ng/L
Propane	25	ng/L
Propene	25	ng/L
i-butane	25	ng/L
n-butane	25	ng/L

7.1 Calculations

The headspace prepared through this procedure can be analyzed to give the vapor, or gas phase concentration of the analytes. That concentration can then be used to calculate the concentration of the analytes in the original water samples. This is done by multiplying the gas phase (vapor units) concentrations by the units specified in Table 7.1 below.

Table 7.1
Conversion Factors

Analyte	Vapor Units	Factor	Units
Carbon dioxide	% by volume	21.3	mg/L
Oxygen	% by volume	4.85	mg/L
Nitrogen	% by volume	4.06	mg/L
Methane	ppmv	0.243	µg/L
Ethane	ppmv	472	ng/L
Ethene	ppmv	525	ng/L
Propane	ppmv	672	ng/L
Propene	ppmv	782	ng/L
i-butane	ppmv	854	ng/L
n-butane	ppmv	865	ng/L

8.0 Safety

The primary concern with this preparation method is the use of sharps. Needles should always be capped when not in use. The proper method for capping a needle is to insert the needle into the cap while the cap is lying on the bench top. Never attempt to cap a needle by holding the cap.

The other safety concern is handling compressed gas cylinders. Cylinders should always be stored upright and chained in place. Cylinders should only be moved using a dolly designed for cylinder transport. Safety glasses shall be worn in the laboratory at all times. Consult Microseeps Chemical Hygiene Plan for more specific information on laboratory safety.

9.0 Waste

Samples are kept for 30 days following analysis. There is no hazardous waste generated during the analytical process. The analysts dispose of the sample vials in the general laboratory trash.

10.0 References

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste. SW-846, 3rd ed., Office of Solid Waste and Emergency Response, Washington, DC. 1986.

Newel, B.S., RSKSOP-175, Sample Preparation and Calculations for Dissolved Gas Analysis in Water Samples using a GC Headspace Equilibration Technique. Revision No. 0, August 1994.

Newel, B.S., RSK-SOP-147, Gas Chromatographic Analysis of Gaseous Samples for Part-Per-Million Levels of Nitrous Oxide, Methane, Ethylene, and Ethane. Revision No. 0, August 1993.

American Society for Testing and Materials, Standard Practice for Analysis of Reformed Gas by Gas Chromatography. Annual Book of ASTM Standards. Vol. 14.02, 1994.

Kampbell, D.H. and Vandegrift, S.A., Analysis of Dissolved Methane, Ethane, and Ethylene in Ground Water by a Standard Gas Chromatographic Technique. Journal of Chromatographic Science. Vol. 36, May 1998.

Microseeps, Incorporated

Standard Operating Procedure For the Analysis of Volatile Fatty Acids in Water

Controlled Copy No. _____

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Technical Director

Date: 11-13-02

1.0 Scope and Application

Method AM21 is used to determine the concentration of selected "volatile fatty acids" (VFAs) in water samples. This method is recommended for use by, or under the supervision of, analysts experienced in sample preparation, the operation of gas chromatographs and in the interpretation of chromatograms.

1.1 Analyte List

Specifically, Method AM21 is used to determine the dissolved concentration of the following anions. In aqueous solutions at low pHs (<4) these anions readily form the corresponding acids. The names of the acids are given in parentheses.

- Acetate (Acetic acid)
- Propionate (Propionic acid)
- Butyrate (Butyric acid)
- Lactate (Lactic acid)
- Pyruvate (Pyruvic acid)
- Hydroxyisobutyrate (Hydroxisobutyric acid)

1.2 Matrix

This method is applicable to samples submitted from groundwater.

2.0 Method Summary

Analysis of VFAs in a water sample is accomplished by an addition of a chromatography aid to the samples followed by direct injection of an aqueous sample into a gas chromatograph (GC) that is equipped with a flame ionization detector (FID). The data are transferred to a microcomputer, converted to digital format, stored, and processed using a chromatography data system.

2.1 Definitions

Not applicable

2.2 Method Limitations

Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. The analyst should demonstrate the absence of carryover contamination by analysis of blank water to which the chromatography aid had been added. This demonstration should be performed prior to the analysis of a sample set and when carryover contamination is suspected (after high samples). In the event that "ghost peaks" (peaks similar to previous

sample) appear when a properly prepared water blank is analyzed, measures should be taken to eliminate the carryover contamination.
Extra peaks in a chromatogram can be actual peaks from a previous run. Contamination from late eluting peaks can occur when the time between successive injections is too short. The analyst should be certain that all peaks have eluted from the previous analysis prior to analyzing any sample or standard. If samples or standard chromatograms contain suspected "extra peaks" the sample should be analyzed again after a clean baseline is established.

3.0 Apparatus, Materials, and Operating Conditions

3.1 Apparatus

- Gas Chromatograph: Varian 3400.
- Autosampler: Varian 8100.
- Injector: Straight flash.
- Detector: Flame Ionization Detector.
- Column: Proprietary.
- Quantification Software: EZ Chrome.

3.2 Materials

- Appropriate standards and reagents.
- Gas tight syringe.
- Volumetric glassware.

3.3 Operating Conditions

- Packed Column Temperature: 175°C.
- Injector and FID Temperature: 210°C.
- Carrier Gas: Nitrogen @ 40 ml/min.

4.0 Reagents

All reagents are purchased as laboratory grade chemicals. Purchased standards are received with Certificates of Analyses. Standards are labeled and documented in accordance with Microseeps Standard Operating Procedure for Standards and Reference Materials SOP-ADM 15.

4.1 Standard Preparation Procedures

4.1.1 Calibration standards are prepared through serial dilution of a mix of the VFAs. Standards are prepared at concentrations of 500, 250, 100, 25, 5, and 1 mg/l.

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4.1.2 All standards and blanks are dosed with a chromatography aid prior to analysis.

4.2 Glassware and Storage Requirements for Reagents and Standards

Reagents can be stored for six months in a glass container. Reagents shall be refrigerated to 4°C.

5.0 Procedure

The following lists the specifics for sample receipt.

Sample Container Type:	Standard 40 ml VOA vial
Holding Time:	21 days
Preservation:	None
Volume:	Two 40 mL vials full with zero headspace.
Storage Requirements:	Ship and store at 4°C ±2°.

Initial Demonstration of Proficiency

Before this analysis can be routinely performed (*i.e.* immediately after any analyst, equipment or equipment location change) an initial demonstration of proficiency must be performed. This is done by analyzing four duplicate samples of a mid-range standard. For each analyte the percent relative standard deviation (% RSD) is calculated by:

$$\%RSD = \frac{\sigma_{n-1}}{S} \times 100\%$$

Where:

% RSD = is the percent relative standard deviation of the response
S = is the mean response (X1+X2+X3+X4)/4

The % RSD must be less than 20% for each analyte. If this criterion is not met, the system is checked and those analytes are to be re-analyzed. If any of those compounds fail the criteria, the system should be re-checked and the initial demonstration of proficiency repeated.

5.1 Sample Preparation

Addition of Chromatography aid: proprietary

5.2 Calibration

5.2.1 Calibration standards with concentrations of 0, 1, 5, 25, 100, 250, and 500 mg/L are used for the initial calibration.

5.2.2 A 1 µl aliquot of each calibration standard and the blank are placed in the autosampler.

Acceptance Criteria: Initial calibration curves meet linearity requirements by having a calculated %RSD equal to or less than 30 and a coefficient of determination greater than or equal to 0.995.

5.2.3 A second source standard, that has all acids present at concentrations near the middle of the calibration curve, is analyzed after each calibration.

Acceptance Criteria: The second source calibration verification must measure each analyte within 75-125 % recovery for the calibration to be considered valid.

5.2.4 Continuing calibration verifications are analyzed after every twenty samples or once per batch, whichever is more frequent. Continuing calibration verifications must have a calculated percent difference less than or equal to 20%.

5.2.5 If the above acceptance criteria cannot be met, the analyst should attempt to recalibrate the instrument. If the acceptance criteria still cannot be met, the Lead Analyst shall be notified immediately.

5.3 Sample Analysis

A sample batch consists of 20 samples or fewer analyzed at a given time. The following procedure is followed for sample analysis.

5.3.1 The sample is briefly uncapped (when dissolved in water the acids are minimally volatile) and a standard portion of the chromatography aid is added, as a solid, to the 40 ml vial.

5.3.2 A few drops of sample are displaced by the addition of the chromatography aid.

5.3.3 The cap is replaced and the vial is shaken.

5.3.4 The vial is loaded into the autosampler with the standards in the sequence specified in the data acquisition system.

5.4 Quality Control

5.4.1 Quality Control Sample Frequency: Laboratory Control Sample, Matrix spikes (MS), matrix spike duplicates (MSD), continuing calibration verifications (CCV) and instrument blanks are analyzed every 20 samples or a minimum of once per analytical batch.

In the event that sample volume does not permit an MS/MSD, an LCS and LCS duplicate are analyzed using the same criteria as for the MS/MSD.

5.4.2 Blank Acceptance Criteria: Instrument blanks may not have any analytes present above the reporting limits specified in Table 7.0

Corrective Action: If this criterion is not met, inspect all glassware, etc. and then prepare and analyze another blank. Blanks should be run until this criterion is met. If three blanks are analyzed in succession and this criterion is still not met, the laboratory director shall be notified.

5.4.3 Matrix Spike and Matrix Spike Duplicate Acceptance Criteria: Experience has shown that poor matrix spike and matrix spike duplicate results most often are attributed to matrix interferences. As such, there are no criteria for these analyses. The results are reported to the customer and percent recoveries outside the 70% - 130% range and percent differences greater than 20% are noted in a case narrative.

5.4.4 Laboratory Control Sample Criteria: Percent recoveries of between 70% and 130%.

5.4.4 Calibration records are generated and stored. All current QA/QC records will be maintained in the laboratory during the course of the project and thereafter as determined by the customer up to a maximum of five years. At the end of each year, all calibration records are stored in the Records Storage Area.

5.5 Capturing and Submitting Data

Measured peak areas are converted to concentrations using known values from known standards. Using a compilation of the standard concentration vs. the response area, a linear, least-squares correlation is performed. The blank is included in the fit and the fitting routine does not force a zero intercept.

6.0 Secondary Data Review

All analytical data must undergo a two-tier review. The data must be reviewed for completeness and accuracy by the analyst and must be checked by the Laboratory Director or his designee before the data can be released as final data.

The analyst is responsible for ensuring that all calibrations, calibration checks, matrix spikes, matrix spike duplicates, duplicates, laboratory control samples, and blanks are within the specifications outlined in this SOP.

Analyst Review. As a data package is generated, the analyst is responsible for the following:

- Verifying that all data keyed or imported into a tabular report format matches the raw data report. This includes the result for each parameter along with any manipulations for dilution or non-standard aliquot used.
- Verifying that all QA/QC data matches the raw data reports and that it meets the acceptance criteria.
- Initialing the report forms to indicate the analyst review is complete.

6.1 Peer Review

Because the amount of this type of data generated by Microseeps currently warrants only one certified analyst, a peer review is not conducted on this data.

6.2 Laboratory Director Review

Once the analyst has completed his review and required corrections are made, preliminary fax results may be provided at the request of the customer, but final results are not to be delivered until the lab director or his designee performs the second tier review.

The Laboratory Director reviews 10% of all laboratory data and calculations. This review includes sample results, quality control acceptance limits, and a review of the level of quality control required for the project.

The Laboratory Director's review consists of the following:

- Spot-checking 10% of any calculations performed.
- Checking that detection/reporting limits are consistent with the method and any modifications required due to dilutions.
- Checking the data for consistency with past analyses and/or expected results.
- Verifying QA/QC data for accuracy and compliance with acceptance criteria.
- Review invoice to ensure that all costs are according to quoted prices.
- Initial the report form to indicate that final review is complete and data is ready for delivery to the customer.

6.3 Performance Evaluation Studies

Performance evaluation studies are not conducted on this method because they are not available from a reputable manufacturer of analytical standards.

7.0 Reporting Limits

MDL studies are performed annually on all analytical methods. The studies are undertaken more frequently if a situation warrants. Reporting limits are always higher than the MDLs.

Table 7.0
Reporting Levels

Acid	Reporting Level (mg/l)
Lactic	25
Pyruvic	10
Acetic	1
Propionic	1
Butyric	1

8.0 Safety

Safety glasses are required in all laboratory areas. For other safety concerns, consult Microseeps' Chemical Hygiene Plan. Material Safety Data Sheets for all compounds used in this procedure are available in Microseeps' Conference Room.

9.0 Waste

All sample waste and laboratory-generated waste shall be treated, handled, and disposed in accordance with Microseeps' Standard Operating Procedure for Waste Disposal.

9.1 Waste Minimization

See Microseeps' Standard Operating Procedure for Waste Disposal for Waste Minimization for this procedure if it is applicable.

10.0 References

U.S. Environmental Protection Agency, 1986. Test Methods for Evaluating Solid Waste, Methods 8000 and 8015, SW-846, 3rd ed., Office of Solid Waste and Emergency Response, Washington, DC.

Microseeps, Incorporated

Standard Operating Procedure for the Collection of Dissolved Gases From Water Using the "Bubble Strip" Sampling Technique at the Well Site

Controlled Copy

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Dominic Nestasie
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Signature of Final Approval

Patrick McLoughlin, Ph.D.
Technical Director

Date: 9-6-02

1.0 Purpose and Application

Method SM9 is used to sample groundwater for dissolved gases by preparing and collecting a gas sample that has concentrations of gases in equilibrium with the concentration of the gases dissolved in the sampled groundwater. The concentration of each of those gases in the gas sample can then be measured and, using those gas phase concentrations, the concentrations of the dissolved gases in the sampled groundwater can be calculated. Sampling in this fashion both pre-concentrates the dissolved gasses and fixes their concentrations by removing them from the biologically active groundwater matrix. Specifically, Method SM9 is used to sample for the following dissolved gasses:

Dissolved Gas	CAS Number
Carbon dioxide	124-38-9
Oxygen	7782-44-7
Nitrogen	7727-37-9
Hydrogen	1333-74-0
Methane	74-82-8
Ethane	74-84-0
Ethene	74-85-1
Propane	74-98-6
Propene	115-07-1
n-Butane	106-97-8
i-Butane	75-28-5

This method is recommended for use by, or under the supervision of, sample technicians experienced in sample collection of both water and gas samples.

2.0 Summary of Method

The collection of the gas sample that has concentrations of dissolved gases in equilibrium with the concentration of those gases in the sampled groundwater, or the bubble strip gas sample, can be accomplished through one of two methods. The first is through a method described by Chapelle et. al. in the paper "Practical Considerations for Measuring Hydrogen Concentrations in Groundwater". The second is through a similar method developed at Microseeps and detailed in the instructions for the Microseeps Gas Stripping Cell.

In either method, the fundamentals are the same: a glass container, whose entire volume is in the flow-path of the groundwater, is continuously filled. Once the container is filled, a small volume of a seed gas is injected into the container and the groundwater is circulated past that gas until the analytes have come into equilibration between the gas and the groundwater. The gas is then removed with a syringe, stored in a 22 mL vial that has previously been filled with 1 atmosphere of pure nitrogen, and sent to the laboratory for analysis.

3.0 Interferences

3.1 The most likely source of "interference" is ambient air. Due to the relatively high concentrations of oxygen and nitrogen, a very small amount of air as a contaminant will dramatically skew the results. The sampler must take great care to ensure that air is flushed from the gas tight syringe before removing the sample from the bubble strip container and that no air has entered the syringe or needle prior to injection of the sample into the storage vial.

3.2 Dissolved hydrogen concentrations are very sensitive to a number of interferences:

- Dissolved hydrogen should only be sampled from wells that have been established for a minimum of three months prior to sampling.
- Dissolved hydrogen should only be sampled from wells that are constructed of PVC or stainless steel.
- Submersible pumps that use any type of electric current below the water level may generate molecular hydrogen and **should not be used**. Similarly, metallic lines used for suspending submersible pumps may generate molecular hydrogen and **should not be used.** Direct driven submersible pumps that are suspended by a non-electrically conductive material may be used.

4.0 Apparatus and Materials

- Sample vials (Supelco, Inc, Bellefonte, PA or equivalent).
- Peristaltic pump (Geotech Series II or equivalent). At low depths, a Bennet submersible pump may be used.
- 250 mL glass gas sampling bulb (Supelco, Inc. Bellefonte, PA, or equivalent) or Microseeps Gas Stripping Cell.
- Gas-tight Syringe, 23 gauge needles and stopcock, all luer-lock (or equivalent) and all warmed to at least 20 C before use.
- Tubing that prevents diffusion of analytes (HDPE hard tubing or Cole-Parmer C-Flex™ soft tubing)

5.0 Sample Preparation and Analysis

The sample preparation and analysis will vary depending upon the type of sampling cell used during the sample collection process.

5.1 Microseeps' Method

When using the Microseeps gas stripping cell and storing the sample in a vial, follow the instructions given in Exhibit 1.

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5.2 Chapelle Method, et al:

When using the Supelco cells and sampling by the Chapelle Method, follow the instructions given in Exhibit 2.

5.3 Quality Control

5.3.1 The gas tight property of the syringe should be verified before sampling is begun. Assemble the luer-lock valve onto the syringe. Leave the valve in the open position and depress the plunger. Lock the valve and try to withdraw the plunger. There should be considerable resistance to the movement of the plunger and it should return to the depressed position, or close to it, once it is released.

5.3.2 If the sample is stored in a glass vial provided by Microseeps, injection of the sample into that vial should represent a creation of approximately 10 psig of pressure inside that vial. If there is very little or no resistance to the injection of the sample into the vial a leak in the vial should be suspected. The vial should be disposed of and the sample recollected.

5.3.3 Due to the nature of the target analytes, there is little chance of cross contamination through equipment carryover. Never-the-less, basic practices of mild sampling equipment decontamination, etc. should be followed. If the Microseeps equipment is used, appropriate decontamination procedures are discussed in Exhibit 1. If other sampling equipment is used, that procedure should be adapted accordingly.

5.3.4 The bubble strip collection apparatus, be it Microseeps' or that supplied by another, contains replaceable septa. While these are not single use septa, the sampler is cautioned against over use. Five piercing of a septum between replacements is a good rule of thumb, but the sampler should look to see if bubbles are continually emerging from the septum when it is placed in contact with the water. If such bubbles are emerging sampling should be halted, the septum should be replaced and sample collection for the well currently being sampled should be repeated.

5.4 Results

The concentrations of analytes in the headspace gas are typically specified in a volume-to-volume ratio (% or PPMV). These concentrations are converted to the original analyte concentration in the water (mg/l for permanent gases, nM/l for hydrogen, and ng/l for light hydrocarbons) using a calculation based on Henry's Law and the ideal gas law.

6.0 Capturing and Submitting Data

Not Applicable

7.0 Reporting Limits

Table 7.0 below gives the reporting limits for analytes that can be detected from samples collected using this sampling method.

Table 7.0
Reporting Limits

Analyte	Reporting Limit	Water Units
Hydrogen	0.3	nm
Carbon Dioxide	0.6	mg/L
Oxygen	0.15	mg/L
Nitrogen	0.4	mg/L
Methane	0.015	µg/L
Ethane	5	ng/L
Ethene	5	ng/L
Propane	25	ng/L
Propene	25	ng/L
i-butane	25	ng/L
n-butane	25	ng/L

7.1 Conversion Factors

Once the vapor sample is collected and stored in the supplied container, it can be analyzed to measure the concentration of the analytes in the vapor/gas. That concentration can be used to calculate the concentration of the analytes in the groundwater. This is done by multiplying the measured gas phase concentrations by the conversion factors given in Table 7.1 below.

Table 7.1
Conversion Factors

Analyte	Vapor Units	Conversion Factor	Water Units
Hydrogen	ppmv	1.98	nm*
Carbon Dioxide	% by volume	36.6	mg/L
Oxygen	% by volume	0.998	mg/L
Nitrogen	% by volume	0.444	mg/L
**Methane	% by volume	524	µg/L
Methane	ppmv	0.0524	µg/L
Ethane	ppmv	136	ng/L
Ethene	ppmv	329	ng/L
Propane	ppmv	151	ng/L
Propene	ppmv	482	ng/L
i-butane	ppmv	121	ng/L
n-butane	ppmv	148	ng/L

* nM (nanomoles per liter) is the industry standard unit for hydrogen.
**High level methane is quantified off of the TCD as % volume.

8.0 Sampling Safety

All sampling shall be conducted in accordance with the site-specific Health and Safety Plan. The specific safety issue with the Bubble Strip Sampling Method is handling needles with care. Needles shall always be capped when not in use and when being handled, especially when being transferred between persons. When placing a cap on a needle it is important to not hold the cap, but place the cap on a solid object and then slide the needle into the cap.

9.0 Waste Disposal

All sampling waste disposal shall be in accordance with the site-specific Work Plan.

10.0 References

Cao, X.L., Hewitt, C.N., Waterhouse, K.S., Determination of Reactive Hydrocarbons by Capillary Gas Chromatography with the Reduction Gas Detector. Journal of Chromatography A. May 1994.
Chapelle, F.H., et.al., Practical Considerations for Measuring Hydrogen Concentrations in Groundwater. Environmental Science and Technology. Volume 31, No. 1 0, pp. 2873-2877, 1997.

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Exhibit 1

Microseeps Gas Stripping Cell

Microseeps Gas Stripping Cell Instructions
INSTALLATION AND OPERATION

To place the gas stripping cell into service:

1. Remove one of the cell assemblies from the packing carton. Refer to Figure 1 to become familiar with the parts of the cell. Make sure that the syringe is at least at 20 °C before proceeding.
2. Connect the inlet tube of the cell to the outlet of your pump. The inlet tube is designed to connect to ¼ inch O.D. hard tubing. Secure the connection (nylon wire ties are recommended).
3. Insert the drain tube of the cell into a waste container, keeping the end of the tube at the bottom of the container. Any waste container of suitable size may be used. A 2-liter soda pop bottle may be placed in the waste container to determine pumping flow rate.
4. Secure the cell assembly so that the housing cover is above the glass housing (*i.e.* upright). A ring stand and clamp are recommended for this purpose.
5. Turn the pump on and check for leaks. If any leaks are found seal them before proceeding. Measure, in ml per minute, the flow rate of the pump. If a 2-liter soda pop bottle was used, the flow rate can be determined by measuring how many minutes it takes to fill the bottle, then substituting the measured time into the following equation.

   Flow = 2000 ml/time to fill in minutes

   Consult Table 1 to determine the equilibrium time needed to gas strip at this flow rate.

   NOTE: Use a flow rate between 100 ml/min and 500 ml/min. Do not turn off the pump.
6. Unclamp the cell assembly, invert it and re-secure the assembly in the inverted position. Make sure the drain tube is still in the waste container and the end of the drain tube is near the bottom of the bottle.
7. Connect the (supplied) stopcock to the syringe and the (supplied) needle to the stopcock. Place the stopcock in the open position (*i.e.* so that the stopcock handle is in-line with the syringe). Draw the plunger back on the syringe to the 20.0-mL mark. Keeping the cell in the inverted position, insert the needle into the needle guide. Pierce the septum and inject the air into the cell. Then remove the needle and syringe from the assembly and carefully cover the needle. Do not discard the syringe apparatus.

   Figures and Tables on Reverse
8. Start timing and let the ground water pump through the cell for the time specified in Table 1 for your particular pumping speed. Meanwhile, be sure that the sample vial is properly labeled and that the flow rate and any other relevant field data are recorded in the field log.

   NOTE: Be sure to keep the end of the drain tube at the bottom of the waste container. This will insure that outside air is not drawn into the cell. Failure to do this will invalidate the sample.
9. When the equilibration time is up, **turn off the pump,** unclamp the cell and reclamp it in its upright position. Verify that the plunger of the syringe is pushed all the way in and that the stopcock is in the open position, then insert the needle into the needle guide and pierce the septum. Insert the needle until the tip is within the gas. Withdraw 1-mL of gas by pulling back on the syringe plunger while holding the syringe body in place. Remove the syringe from the cell and expel the sample. Immediately re-insert the needle into the needle guide and pierce the septum. Withdraw a 15-mL sample of gas and, with the needle still through the septum, close the stopcock. Immediately proceed to the next step.
10. If the sample is to be stored and shipped to the fixed laboratory, rapidly withdraw the needle from the septum and place it through the septum on the sample vial (see Figure 2). Open the stopcock and completely depress the syringe barrel. If the sample is to be analyzed in the mobile laboratory, cork the needle tip by placing it into a septum.
11. When done, discard the syringe apparatus according to Local, State and Federal regulations.

Decontamination/Cleaning

Pump at least 1 liter of potable water through the cell. The cell assembly is now ready for re-use.

The only expendable part of the cell is the sampling septum (part 7). Normally, each septum may be used for the collection of up to 5 samples. If bubbles are seen rising up from the septum when the cell is inverted the septum MUST be replaced. Instructions for replacing the septum are provided on the reverse.

SAMPLING QUESTIONS?

CALL MICROSEEPS AT **1-412-826-5245**
MON.- FRI.9 TO 5 PM EST

Figure 1. Cross section of Microseeps Gas Stripping Cell

1. Housing Cover
2. Jet Spray Nozzle
3. Nylon Tie
4. Inlet Tube
5. Needle Guide Port
6. Drain Tube
7. Replaceable Septum
8. Glass Housing

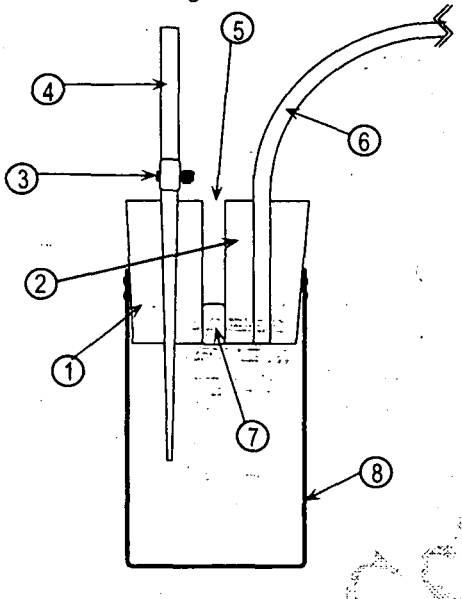

Figure 2. Cross section of septum bottle

1. Septum
2. Metal Closure
3. Glass vial

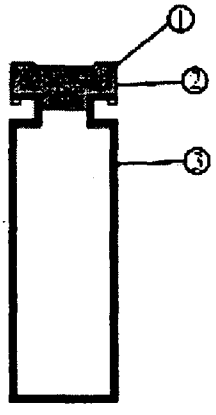

Table 1.

Flow rate (ml/min)	Sampling time (min)
100-120	30
130-150	25
160-200	20
210-300	15
>300	10

Replacing the Sampling Port Septum

All part numbers refer to Figure 1.

1. **Remove the housing cover (part 1) from the glass housing (part 8).**
2. **Use a handy, blunt tipped object to push the replaceable septum (part 7) out of the housing cover. The cover to a needle works well for this purpose, but be sure that the needle is NOT in the cover. Discard the old septum.**
4. **Take a new septum and wet both the new septum and the housing cover with potable water.**
5. **Carefully using the same blunt instrument used in step three above, slide the new septum into the hole from which the old septum was removed. The bottom of the new septum must be flush with the narrow end of the housing cover.**
6. **If the housing cover is not still wet, wet it again with potable water. Place the bottom end of the housing cover into the glass housing and push it in until less than 3/8" are above the rim of the glass housing. This may require some force.**
7. **Follow the cleaning procedures described above to prepare the cell for a return to service.**

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Exhibit 2

Chapelle Sampling Method

- After the well is purged connect the gas-sampling bulb, or bubble strip cell, to the well and fill the cell completely with water, eliminating all headspace and bubbles. Continue to pump with the cell mounted at a 30° angle downward from the inlet to the outlet. Pump three cell volumes through the cell at 300-600mL/min. While continuing to pump, inject 25mL of either laboratory supplied inert gas or ambient air through the septum port at the inlet end of the cell. Pump an additional 30 min.

- Using a gas-tight, locking syringe that has been filled and purged with either the laboratory supplied inert gas or ambient air, withdraw 12mL of headspace gas from the flow through cell into the syringe. Lock and remove the syringe needle from the septum and contain the sample in one of the following ways.

- If the sample is to be analyzed in the on-site laboratory, plug the needle by inserting it into a rubber septum that has been provided by Microseeps. Continue to pump an additional five minutes and, using a second needle, collect a second aliquot of the gas and use a second septum to plug the needle. After the duplicate sample has been collected, both samples should be immediately transported to Microseeps onsite lab for analysis.

- If the sample is to be analyzed in Microseeps fixed laboratory in Pittsburgh, PA, a 10 cc portion of the sample is injected into a sample vial provided by Microseeps. After pumping for an additional 5 minutes, a duplicate sample is taken and contained in a second sample vial using the procedure just described. To minimize the potential for leakage, care should be taken to insure that the syringe needle is plunged squarely into and withdrawn squarely from the sample vial so as not to tear the septum. Both sample vials should be labeled and shipped to Microseeps for analysis at:

Microseeps Incorporated
220 William Pitt Way
Pittsburgh, PA 15238